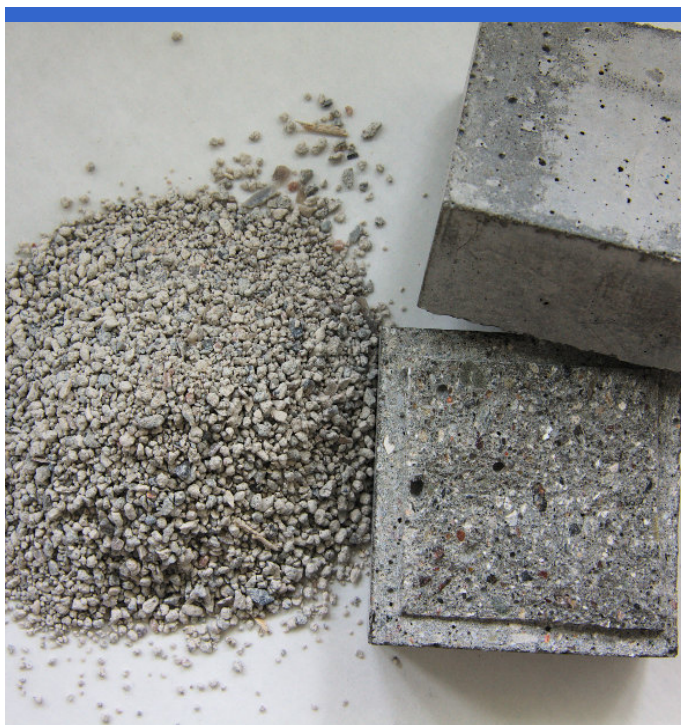# Concrete waste as internal water reservoir in high strength concrete

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Master thesis

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Preface

This report is the final thesis for the master education in Civil Engineering at the Technical University of Denmark. The project has been carried out from January 4th to June 17th, 2016. The scope of the report is 35 ECTS points.

The supervisor on the thesis is Professor Lisbeth M. Ottosen.

Leander Jørgensen,
Lyngby, Denmark
17. June, 2016

Abstract

Autogenous shrinkage is a shrinkage phenomena in concrete, caused by self-dessication and occurs mainly in high strength concrete. The phenomena has been extensively studied over the last couples of decades. Solutions to the problem is mainly the use of super absorbent polymeers or wet aggregates functioning as an internal curing agent. LECA is a material that has been tested extensively for the use as an internal curing agent and performs well in mitigating autogenous shrinkage. This project investigates whether it is possible to use crushed concrete waste as an internal curing agent. The motivation for this is that in contrast to the production of LECA the reuse of concrete is a less energy demanding process. The current use of concrete waste in Denmark is using it as hard rock under roads, this project seeks to find if more value can be added to the material.

Crushed concrete waste was acquired from RGS 90. Origin of the concrete waste is not traced and therefore unknown to the author. The fraction of the waste product passing the 2 mm sieve and retained on the 0.5 mm sieve was used for testing and for mixing in the concrete. The particle density, particle size distribution and loss on ignition was measured. The measured particle density on $\rho_{\text{waste product}} = 2335 \pm 11\text{kg/m}^3$ results in an estimated sand-mortar ratio on $\alpha \in \{0.32 - 0.41\}$ based on table-values. Furthermore placing the aggregate as normal weight aggregate. Considering the particle size distribution, the waste product contained larger particles than the sand it replaced. However the elongated form of the waste product particles made the particle size distribution challenging to determine precisely and the form of the particles might affect the workability negatively. Pieces of wood was noticed in the waste product, however loss on ignition was measured to $\text{LOI} = 3.0 \pm 0.2\,\%$ which lies within the requirements in the american standard. The strength was seen to be unaffected by the addition of wet waste product to the mortar. Soaking the waste product in coloured water did not affect the colour of the mortar when opened 14. days after casting. Desorption curve on the waste product was determined and found to be less optimal than LECA, and it did not release enough water in regard to the demand in the American standard. Absorption proved difficult to determine and one method yielded $A_{24h,\text{WP,pan}} = 7.79 \pm 0.05\,\%$ while another method yielded $A_{24h,\text{WP,pycno}} = 1.68 \pm 0.18\,\%$. Finally, shrinkage measurements using a dilatometer did not show any effect of adding the waste product on reducing shrinkage.

Overall no evidence has been found in this project that the concrete waste from this batch is suitable for use as internal water reservoir in high strength concrete. Further research could test other batches, maybe beneficially from known sources, if possible.

Resumé (Danish)

Autogent svind er et fænomen forårsaget af selvudtørring i beton og er primært et problem i højstyrkebeton. Der har været foretaget mange studier af fænomenet over de sidste par årtier. Løsningerne er primært brugen af super absorberende polymerer og våde aggregater der fungerer som interne vandreservoirer i betonen. LECA er et materiale der har været grundigt testet og som har gode egenskaber til at forhindre autogent svind. Dette projekt undersøger om det er muligt at anvende nedknust affaldsbeton som interne vandreservoirs i højstyrkebeton til at undgå autogent svind. Motivationen er at i modsætning produktionen af LECA er produktionen af nedknust betonaffald mindre energikrævende. Den nuværende brug af beton affald i Danmark er som fyld under veje. Dette projekt ønsker at undersøge om det er muligt at øge værdien af beton affald ved at finde anvendelsesmetoder der tilføjer mere værdi til materialet.

Nedknust beton affald blev afhentet på RGS 90. RGS 90 sporer ikke oprindelse af betonaffaldet og den er derfor ukendt. Den fraktion af affaldsproduktet der arbejdes med i dette projekt er den der passerer 2 mm sigten men bliver holdt tilbage på 0.5 mm sigten. Partikel densitet, kornkurvefordeling og glødetab blev målt. Partikel densiteten på $\rho_{\text{waste product}} = 2335 \pm 11 \text{kg/m}^3$ resulterer i et estimat af sand-mørtel forholdet på $\alpha \in \{0.32 - 0.41\}$ baseret på tabelværdier fra litteraturen. Yderligere placerer densiteten affaldsproduktet i kategorien normalvægts aggregater. Kornkurvefordelingen blev testet både på sand og affaldsproduktet og viste at affaldsproduktet bestod af større partikler end det erstattede sand. Målingen blev dog besværliggjort af affaldsproduktets aflange form. Den aflange form kan påvirke bearbejdeligheden af den flydende beton negativt. Affaldsproduktet indeholdt mindre stykker træ. Glødetabet på $\text{LOI} = 3.0 \pm 0.2\,\%$ ligger dog indenfor kravene i den amerikanske standard. Styrken var upåvirket af tilføjelsen af affaldsproduktet. Der blev ikke iagtaget nogen farveændring af mørtelprøver åbnet 14. dage efter støbning, når der blev iblandet affaldsprodukt vandmættet med farvet vand. Desorptionskurve for affaldsproduktet blev bestemt og var mindre optimal end desorptions kurven for LECA, den levede desuden ikke op til kravene i den amerikanske standard. Målingen af absorption skabte udfordringer. En metode målte absorptionen til $A_{24h,\text{WP,pan}} = 7.79 \pm 0.05\,\%$, mens en anden metode målte absorptionen til $A_{24h,\text{WP,pycno}} = 1.68 \pm 0.18\,\%$. Direkte måling af autogent svind ved brug af et dilatometer viste ikke nogen effekt af at iblande affaldsprodukt.

Generelt fandt projektet ingen bevis for at betonaffald fra den undersøgte batch er anvendelig til at undgå autogent svind i højstyrkebeton. Videre studier kan fokuserer på at undersøge andre batches, med fordel af kendt oprindelse, hvis muligt.

Contents

Structure of report

This report consists of four main sections.

Introduction In this section the background and aim of the project is presented. Relevant literature and theory is outlined as well.

Materials and Methods In this section the materials and the test methods used in this project is described. Formulas used for processing the data is also included in this section.

Results and Discussion In this section results are presented and discussed immediately. Results are presented as graphs, tables or numbers. If more than two measurements have been made results are given as the mean value $\pm$ one standard deviation $X = \mu \pm \sigma$.

Conclusion Outlines the conclusions in this project

Raw measurement data are available in appendix.

Comma is used as thousand delimiter and period is used as decimal delimiter, in accordance with the British system.

The waste product will be abbreviated as WP through the report.

1 Introduction

1.1 History of concrete

Concrete has been used extensively since its *redevelopment*[1] in the 19th century by primarily L. J. Vicat (1786–1861), Joseph Aspdin (1779–1855) and Isaac Charles Johnson (1811–1911) (Herholdt et al., 1985). Through the 20th century concrete became the most used material for construction (Mehta and Monteiro, 2014). Its versatile use includes houses, flats, harbours, skyscrapers and bridges. According to U.S. Geological Survey (2016) 4,100 millions metric tonnes of cement was produced world wide in 2015. High performance concrete (HPC), characterized by high strength and being more dense than ordinary concrete, were first developed in the 1980s (Jensen and Hansen, 2001). Where normal strength concrete typically consists of cement, water and aggregates. High performance concrete also consists of different admixtures, like super-plasticizers, in order to get mixtures with low w/c-ratios that are still workable.

1.2 Autogenous shrinkage

The hydration of cement is water consuming, see Table 1.1. Lowering the water-cement

Table 1.1: Hydration of cement, (Mehta and Monteiro, 2014)

Reaction of..		
C_3S		$2\,C_3S + 6\,H \longrightarrow C_3S_2H_3 + 3\,CH$
C_2S		$2\,C_2S + 4\,H \longrightarrow C_3S_2H_3 + CH$
C_3A	Gypsum not present:	$C_3A + 6\,H \longrightarrow C_3AH_6$
	Gypsum present: Step 1	$C_3A + 3\,CSH_2 + 26\,H \longrightarrow C_3A \cdot 3\,CS \cdot H_{32}$
	Gypsum present: Step 2	$C_3A + 3\,CSH_2 + 26\,H \longrightarrow C_3A \cdot 3\,CS \cdot H_{32}$
C_4AF		Similar reaction to C_3A

ratio introduces therefore more challenges than only loss of workability. Lowered enough,

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[1]At least materials very similar to modern day concrete has been around for thousands of years. The Romans are famous for using concrete for their aqueducts and especially for Pantheon and Colosseum.

typically below a water cement ratio of 0.4 (Kovler and Jensen, 2005), problems with self-dessication becomes an issue. It is self-dessication that leads to autogenous shrinkage. In order to understand the concept it helps to imagine a pore in the pore system of the newly cast concrete. To start with the pore is filled with water, however more and more water is being used by the hydrating cement. So some time later in the process the water in the pore is almost used up. The remaining water in the pore begin to form a meniscus. Due to surface tension this forming of menisci in the pore system introduces compressive pressure in the concrete material. The shrinkage caused by this compressive pressure is called autogenous shrinkage. According to Jensen and Hansen (2001) this can lead to micro-cracks that might both affect strength and durability.

Several studies find that autogenous shrinkage can be mitigated, and even completely prevented, by using internal curing ((Bentur et al., 2001), (Lura et al., 2014), (Suzuki et al., 2009)). Kovler and Jensen (2005) presents three ways of performning internal curing in concrete. One option of internal curing is by using internal sealing with water-soluble chemicals, where the sealing hinders the loss of water during hardening. In high-strength concrete where autogenous shrinkage is a problem because of self-dessication, sealing does not solve the problem. The two alternative options is to use either wet aggregates or super-absorbent polymers (SAP) as internal curing agents. When it comes to wet aggregates Kovler and Jensen (2005) only mentions light-weight aggregates (LWA) however a recent study by Zou et al. (2015) found that it is possible to use normal-weight aggregates (NWA) as well. Since this study focuses on the use of pre-soaked aggregates as internal curing agents, the benefits of using super-absorbent polymers will be left for the reader own investigation. The principle of internal curing is sketched in Figure 1.1. Prior to mixing the concrete, the internal curing agent is

| (a) | (b) | (c) |
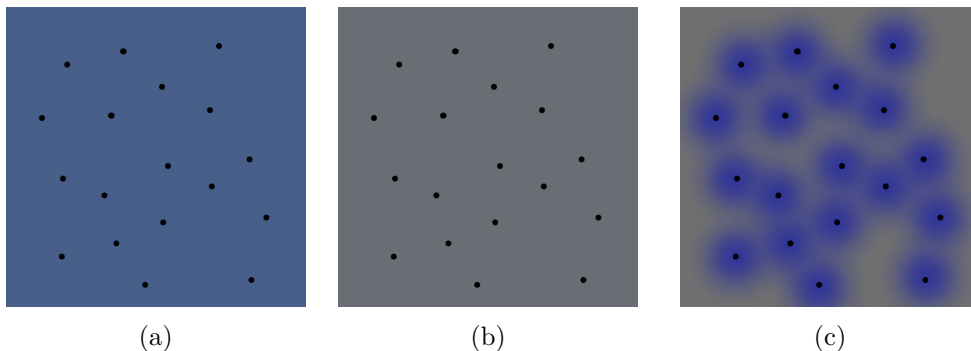
Figure 1.1: Principle of internal curing using wet lightweight aggregates. a) $t = 0$ freshly mixed concrete, black dots indicate internal curing agent; b) $t = \Delta t$ Water is consumed during hardening; c) $t = \Delta t + \Delta t_2$ When the relative humidity gets low enough, the internal curing agent release their water content

pre-soaked in water. A percentage of the sand-fraction is replaced with the internal curing agent and all the ingredients is mixed together. Early in the process, when the humidity in the concrete is still high, the internal curing agent holds the curing water

back. When the humidity in the concrete decreases the curing agent will begin to release the curing water. Thereby delivering the curing water at a time when it is needed to mitigate self-dessication, but so late in the process that it does not affect the strength. In principle it resembles external curing, which does not work here since the water will not penetrate deep enough when only applied on the surface.

1.3 Aim of this project

The fundamental question this study seeks to answer is this:

Can concrete waste function as an internal water reservoir in high strength concrete?

In order to answer this the basic properties for internal curing needs to be investigated for waste concrete. These investigations will be compared to results from other studies on other materials and with ASTM C1761/C1761M:2015. An American standard is used since no European or Danish standard on internal curing is known to the author.

1.4 Concrete waste

According to Miljøstyrelsen (2015) 680,664 tonnes pure[2] concrete waste was registered in Denmark in 2013. According to the same source this number is probably half the real number since not all concrete waste is registered. 97 % of the registered concrete waste was reused in 2013. Currently the waste concrete is primarily reused as the hard core under roads. Even though 97 % is a fairly high percentage of reuse, it is worth considering the value of the product it replaces. Miljøstyrelsen (2015) notes that the use of concrete waste over gravel for road construction leads to a higher carbon footprint. Considering gravel is an readily available resource, it might be desirable to find alternative reuse methods for concrete waste, so it can be reused more effectively. The motivation for investigating alternative ways of reusing concrete waste, stems therefore not from a urgent need to solve a waste-problem. More so from an interest in finding alternative reuse methods that might add more value to the material and so may assist society in solving bigger problems.

Research on the reuse of concrete waste for other purposes has mainly focused on the reuse of aggregates. Safiuddin et al. (2013) reviews the research on reuse of concrete aggregates. Generally reused aggregates performs slightly worse than virgin aggregates on nearly all measured parameters, however the authors conclude the reusing of aggregates have a significant economic and environmental potential. In Denmark reuse of aggregates is not common practice, however according to Miljøstyrelsen (2015) it is common practice in Switzerland, Germany, The Netherlands, Belgium and Japan.

Miljøstyrelsen (2015) includes a few examples of direct reuse of concrete structures, like the Gemini residence on Islands Brygge in Copenhagen where old concrete silos

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[2]Other waste fractions also contains concrete waste e.g. contaminated with bricks, tiles and ceramics

have been converted into apartments. Miljøstyrelsen (2015) do also consider the reuse of concrete elements in new construction projects, but have no examples of such practice. Miljøstyrelsen (2015) notes that the way we design buildings today, will probably not leave the materials usable in 50-100 years time. So in order to do a more direct reuse of concrete elements, there will be a demand for thinking about it in the design process.

Direct reusing of concrete structures or elements will not leave material for internally curing agents as investigated in this project. However reusing aggregates or using the concrete as filler under the roads might actually be possible combined with using part of the materials for internally curing. In reusing aggregates and filler the aggregates is most interesting since this is often the strongest part. However for internal curing the interesting part is the mortar since this is probably more porous and absorbs more water than the aggregates.

1.5   Prior research on internal curing agents

There has been a lot of research on the use of internal curing in order to mitigate autogenous shrinkage. In this regard Ghourchian et al. (2013) investigates the properties of expanded clay (LECA) and zeolite. Expanded clay as internal water reservoir agent has been investigated intensively with (Bentur et al., 2001), (Ghourchian et al., 2013) and (Castro et al., 2011) among others. Castro et al. (2011) investigates expanded clay as well as expanded shale. Lura et al. (2014) investigates the properties of lightweight aggregate produced from biomass-derived waste; Kovler et al. (2004) investigates pumice from volcanic areas in Greece and Iceland. An overview of the literature is available in Table 1.2.

1.6   Working with internal curing

The principle of working with an internal curing agent is to soak it in water for a certain amount of time, remove excess water and mix it into the concrete mixture. The method is relatively simple, however two challenges arises. 1) How to remove excess water? The ideal solution is to reach surface dry condition before mixing. Since if there is free water on the surface of the internal curing agent this will change the water-cement ratio of the mixture. So either surface dry condition needs to be established or the amount of excess water needs to be determined. 2) How much of the internal curing agent is enough? Bentz and Snyder (1999) answers this question by giving the following equation.

$$V_{\text{WP}} = \frac{V_{\text{water}}}{S \cdot p} \tag{1.1}$$

Where:

V_{ICA}:   Volume fraction of sand that should be replaced by the internal curing agent

V_{water}:   Volume of water consumed due to chemical shrinkage, see equation 1.2

S: Saturation of the internal curing agent (0–1)

p: Porosity of the internal curing agent (0–1)

Table 1.2: Short overview of litterature on internal curing agents

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Reused aggregate

Maruyama and Sato (2005) has made preliminary investigations showing the material has potential. However no other studies on reused aggregates has been found.

Expanded clay

Expanded clay, also well known under the commercial name LECA, has been intensively studied for use as internal water reservoirs. The material is generally found to have good absorption and desorption properties for use as internal water reservoir (Ghourchian et al., 2013). $A_{24\text{h}} = 8.9\%wt$(Bentur et al., 2001). $A_{24\text{h}} = 11.00\%wt$ (Ghourchian et al., 2013). Castro et al. (2011) measures absorption in the range of $A_{24\text{h}} = 15\%wt$ to $A_{24\text{h}} = 31\%wt$. Desorption curve is steep, and most of the water is desorped above 97 % RH(Ghourchian et al., 2013). LECA is generally considered being a good internal curing agent.

Pumice

Kovler et al. (2004) investigates pumice from Yali in Greece and Hekla in Iceland. The absorption is determined to lie within 13–27 % from Yali and 32–71 % from Hekla. Generally Kovler et al. (2004) find that pumice has great potential for internal curing.

Expanded shale

Castro et al. (2011) measured 24 absortion values between 10 and 19 %. The same study finds the desorption curve to be satisfiable.

Zeolite

Ghourchian et al. (2013) finds this unsuited for internal curing since the absorbed water is bounded in small pores (Ghourchian et al., 2013). The desorption curve is flat at high relative humidities, where the material retains the absorbed water. The absorption is $A_{24\text{h}} = 15.60\%wt$ and is higher than LECA(Ghourchian et al., 2013).

NWPA - Normal Weight Porous Aggregate (Clay and mud)

Zou et al. (2015) notes that a problem with using light weight aggregates for internal curing, is that they will have a tendency to flow upward in the wet concrete — especially during vibration. Zou et al. (2015) used a mix of red mud and knar clay to produce a NWPA. The created NWPA had an absorption $A_{24\text{h}} = 23.6\%wt$. Zou et al. (2015) concluded that the NWPA performed less efficient than LECA, but was still able to function as an internal curing agent.

Biomass-derived waste

Lura et al. (2014) investigates the possibility of using biomass-derived waste as an internal curing agent. Lura et al. (2014) finds water absorption to be 14.6 and 17.7 % when performed under vacuum. The material have an optimal desorption curve for internal curing, losing most of its water above 97 %. Measurements of autogenous shrinkage shows a clear effect in the study.

Ceramic waste

Suzuki et al. (2009) finds porous ceramic waste to work well as an internal curing agent. The study finds an absorption value on 9.3 %

Instead of using the saturation and porosity, the absorption has been used in this project instead, so that $S \cdot p = A$. The volume of water consumed due to chemical shrinkage can according to Bentz and Snyder (1999) be calculated by

$$V_{\text{water}} = \frac{C_f \cdot CS \cdot \alpha_{\max}}{\rho} \tag{1.2}$$

Where:

C_f:	Cement content in $\frac{\text{kg cement}}{\text{m}^3 \text{ concrete}}$
CS:	Chemical shrinkage during hydration, Bentz and Snyder (1999) states that it is typically 0.06 kg, and this value has been used here.
$\alpha_{\max}$:	Maximum degree of hydration, estimated by $\frac{w/c}{0.40}$
ρ:	Density of mixing water

2 Materials and methods

2.1 The waste product

RGS 90 A/S is a Danish waste handling company who receives construction waste
and treat it for reuse. Crushed concrete waste was collected from RGS 90 A/S on
15. January, 2016. The concrete waste sampled was crushed at RGS 90, their normal
procedure for preparing it for use as filler under roads. Pictures taken on the day of
sampling is available in Figure 2.1. The origin of the concrete waste is not traced,
however the waste is sorted into three fraction: Pure concrete waste, Asphalt concrete,
and concrete waste contaminated with bricks and tiles. The material used in this project

(a) Concrete waste as received at RGS 90

(b) Fresh concrete waste at RGS 90

(c) Concrete waste after crushing

Figure 2.1: Pictures from sampling of concrete waste at RGS 90 (15. january 2016)

consist of crushed mortar and aggregates sampled from the fraction containing only pure concrete. Hereafter called the waste product and abbreviated WP.

2.2   Overview of lab tests

Table 2.1 gives an overview of the tests carried out in this project. In the following sections the methods will be explained in more detail.

Table 2.1: Overview of test carried out

Test	Standards or method
Particle density	DS/EN ISO/TS 17892-3:2004
Particle size distribution	Laser diffractometry combined with sieves
Loss on ignition	DS/EN 196-2:2005
Compressive Strength	Rect. prisms ($4 \times 4 \times 16$ xm)
Colour propagation test	Rect. prisms ($4 \times 4 \times 16$ xm)
Desorption	DS/EN ISO 12571:2013
Absorption	Pan and pycnometer
Autogenous shrinkage	ASTM C1698-09:2014

2.3   Preparation process

The method used for preparing the sampled concrete waste prior to testing and casting is described in Figure 2.2.

The process goes as follows: (i) the waste product was sieved manual through a 4 mm sieve, (ii) then the sieved product ($< 4\,\text{mm}$) was washed with distilled water on a 0.5 mm sieve. (iii) The product retained on the sieve (0.5–4 mm) was collected in a glass container, weighted and put in an oven at $105\,^\circ\text{C}$. (iv) It was kept in the oven until the mass change over a period of 24 h was less than 0.1 % of the wetted mass. (v) The product was placed in a sieve tower with sieves 0.5, 1.0 and 2.0 mm (vi) The product retained on the 0.5 mm sieve and the 1.0 mm sieve was collected and used for further testing..

2.4   Casting of mortar samples

Two types of samples was casted. For autogenous shrinkage samples was casted in corrugated plastic tubes. The rest of the mortar samples was casted in cubic forms measuring $50 \times 50 \times 50$ mm.

Sieve on 4 mm sieve by hand

⬇

Wash sieved product (< 4 mm) on 0.5 mm sieve

⬇

Oven dry at 105 °C until $\Delta m_{24\,h} < 0.1\,\%$-wetted mass

⬇

Sieve through sieve tower of:
2.0 mm
1.0 mm
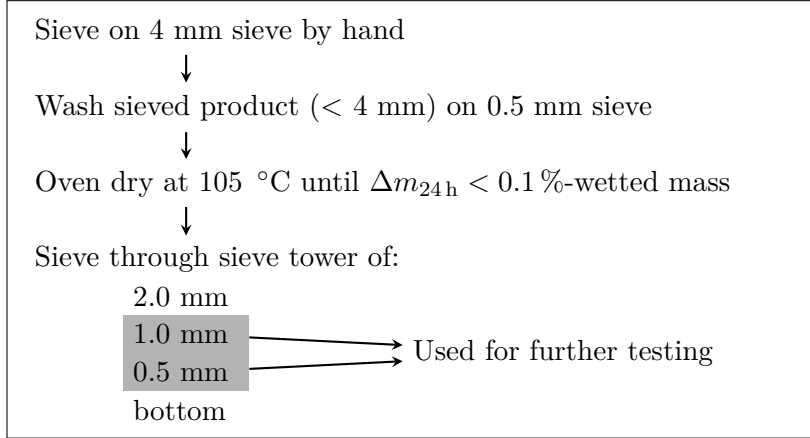0.5 mm   ⟶ Used for further testing
bottom

Figure 2.2: Preparation process

Table 2.2 contains information on the mixing recipes used in this project. The reference recipe was made partly by using experience from concrete proportioning, partly by inspiration from literature, but most importantly by trying different mixtures in the lab. More details on the procedure is available in appendix C. Logbook from casting is available in appendix D with the resulting recipes for each sample. Table 2.3 contains

Table 2.2: Reference mixing recipes (1 % air content assumed)

1 m^3 concrete

	Cement [kg]	Water [kg]	Sand [kg]	Super Plasticiser [kg]	w/c
Cubic samples	698	235	1439	7.0	0.34
Autog. samples	805	27	1237	4.8	0.34

Scaled down to the respectively moulds

	Cement [g]	Water [g]	Sand [g]	Super Plasticiser [g]	w/c
Cubic samples	349	118	719	3.5	0.34
Autog. samples	403	139	618	2.4	0.34

information on the samples used in this project

Challenges encountered during casting

Several issues were encountered and dealt with during casting. The first casting was used to create a workable mortar recipe. Mainly by adjusting sand and superplasticizer. Too much superplasticizer has a tendency to increase segregation as can be seen on the photo in Figure 2.3. At the second casting the workability of the reference recipe developed

Table 2.3: Overview of cast samples

| Sample	Used for	WP % sand replacement	w/c
VII	28 days compressive strength	0 %	0.34
VIII	28 days compressive strength	22 %	0.34
IX	28 days compressive strength	32 %	0.34
X	28 days compressive strength	42 %	0.34
XI	Colour propagation test	22 %	0.34
XII	Colour propagation test	22 %	0.35
XIII	Colour propagation test	32 %	0.35
XIV	Colour propagation test	42 %	0.35
XV	Autogenous shrinkage	0 %	0.34
XV	Autogenous shrinkage	0 %	0.34
XV	Autogenous shrinkage	43 %	0.34
XV	Autogenous shrinkage	43 %	0.34
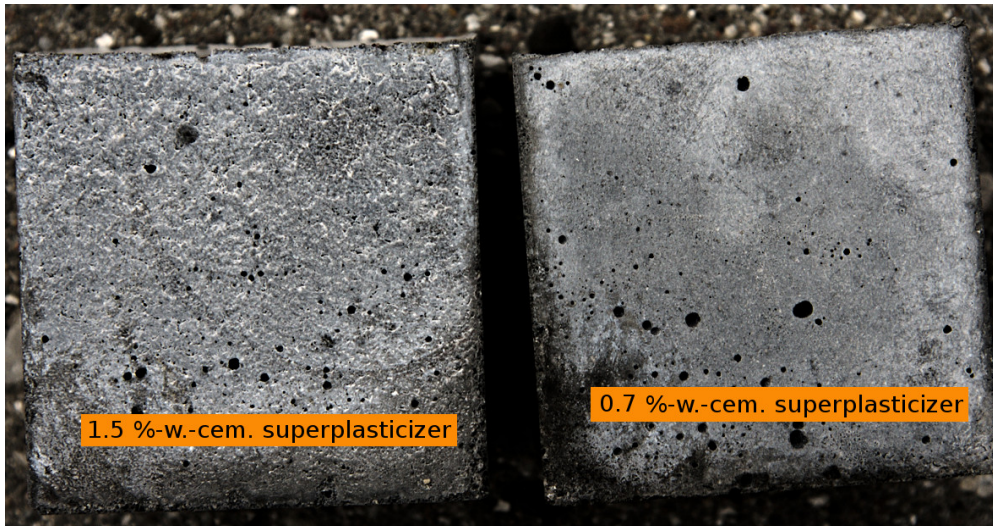
Figure 2.3: Picture of mortar sample surfaces on two different mixtures. The samples are viewed from the side. The contrast has been enhanced in the picture so the effect is more visible.

was different. The most plausible reason must be either change in water content of the sand or because of weighing errors or a combination of the two. For every consecutive casting the sand was dried out in an oven until a couple of hours before casting. Before casting it was left to cool to room temperature under cover.

Furthermore reaching surface dry condition of the waste product is important if the excess water is unknown. During the first casting of samples with waste product the workability changes significantly, indicating that more water was added to the mix than intended. In this project surface dry condition was achieved by heating the waste product on a pan. When the surface dry condition was reached the heat was turned off and the material was stored in a sealed glass container while cooling down.

Particle density

Particle density was determined in accordance with DS/EN ISO/TS 17892-3:2004. Approximately 2 g of oven dry sample was placed in a pycnometer. The weight of the dry sample, pycnometer and plug, M_2 was measured to an accuracy of ± 0.0001g. The temperature of distilled water without entrapped air, was measured, T_3, to an accuracy of $\pm 0.25\,°C$. The pycnometer was filled with the distilled water. The pycnometer was dried on the outside by using a paper cloth. The weight of sample, pycnometer, plug and water, M_3 was measured to an accuracy of ± 0.0001g.

Calibrated pycnometers were used, and the following data was known when the test was conducted: The weight of pycnometer and plug, M_0, with an accuracy of ± 0.0001g; the mass of pycnometer, plug and water, M_1, with an accuracy of ± 0.0001g; the temperature at calibration T_1 to an accuracy of $\pm 0.25\,°C$;

The density of water, $\rho_{w;1}$ at the calibration temperature and the density of water at testing $\rho_{w;3}$ was determined using table values. The particle density was determined by Equation 2.1

$$\rho_s = \frac{M_4}{\frac{M_1 - M_0}{\rho_{w;1}} - \frac{M_3 - M_2}{\rho_{w;3}}} \tag{2.1}$$

Where:
M_0:	Mass of pycnometer + plug (empty, at calibration)
M_1:	Mass of pycnometer + plug + water (at calibration)
M_2:	Mass of pycnometer + plug + sample
M_3:	Mass of pycnometer + plug + sample + water
$\rho_{w;1}$:	Water density at calibration
$\rho_{w;3}$:	Water density at test

By comparing the particle density with table values for the density of mortar and sand, the ratio of sand and mortar in the sample was estimated by Equation 2.2

$$\alpha \equiv \frac{m_{\text{sand}}}{m_{\text{mortar}}} = \frac{\rho_s - \rho_{\text{mortar}}}{\rho_{\text{sand}} - \rho_{\text{mortar}}} \tag{2.2}$$

Where:

α:	sand-mortar ratio
m_{sand}:	Mass of sand (unknown)
m_{mortar}:	Mass of mortar (unknown)
ρ_s:	Particle density (measured)
ρ_{sand}:	Density of sand (table value)
ρ_{mortar}:	Density of mortar (table value)

2.5   Particle size distribution

Particle size distribution on sand was performed by sieving through a 2 mm sieve. The material passing through and the material retained was weighted. The material smaller than 2 mm was mixed with sodium pyrophosphate and ran through laser diffractometry.

Particle size distribution for the waste product was performed by sieving through a 1 mm sieve. The material passing through and the material retained was weighted. The material passing through was used for dry laser diffractometry.

2.6   Loss on Ignition (LOI)

3 crucibles was heated in an oven at 550 °C for 30 minutes. After cooling down for 20 minutes in a desiccator the weight of the empty crucibles, $m_{\text{cruc.}}$ and the weight of the crucibles with the dry material was measured, $m_{\text{cruc.+WP}}$. The crucibles were placed in the oven again for 2 hours. After cooling down for 20 minutes the weight, $m_{\text{cruc.+WP\_Ignited}}$, was measured. The crucibles was handled using a plier.

$$\text{LOI\%} = \frac{m_{\text{cruc.+WP\_Ignited}} - m_{\text{cruc.}}}{m_{\text{cruc.+WP}} - m_{\text{cruc.}}} \cdot 100\% \tag{2.3}$$

2.7   Colour propagation test

1.5 bottle red fruit colour dye was mixed with 1.5 l of distilled water, stirred. The waste product was left for 24 hours to soak in the coloured water. Surface dry condition was achieved by heating the waste product on a pan. The waste product was cast into cubic mortar samples. After 14 days the samples was opened by cutting a 0.5 cm groove all the way around the middle of the sample, and thereafter they were knocked open using a chisel. This was done in order not to affect the colouring.

2.8   Uniaxial compressive strength

Uniaxial compressive tests was carried out on 50 mm cubic samples. Loaded with 1 kN/s in a UCS test machine. The samples were supported on the complete surface area

in top and bottom. In accordance with the machine specification 4 kN was subtracted from the measured load.

2.9 Desorption

Desorption was determined by following the desiccator method described in DS/EN ISO 12571:2013. The waste product was left to soak in distilled water for 24 h $\pm$ 0.5 h. The water was removed by placing the waste product on a 0.5 mm sieve. In order to obtain surface dry conditions the surface of the particles was dried using a fine paper cloth. 5 desiccators was prepared containing solutions of: NaBr (59 %RH); KCl (85 %RH); KNO$_3$ (94.5 %RH); K$_2$SO$_4$ (97.9 %RH); and H$_2$O (100 %RH) respectively. Approximately 15 $\times$ 10 g of the waste product was placed in 15 glass cups with lid. Weight was measured and the cups was placed in the desiccators, 3 in each. The lid were removed from the cups and the pressure in the desiccators were lowered to just above the vapour pressure. The weight of the samples was measured each 24 h $\pm$ 0.5 h until the mass change over 24 hours was less than 0.1 % of the total mass over 3 consecutive measurements. When the experiment ended the samples were dried in an oven at 105 °C until constant mass and the dry weight was measured.

2.10 Absorption

Two methods was used to determine absorption of the waste product.

The pan method

The waste product was dried in an oven at 105 °C until constant mass. The waste product was soaked in distilled water for 24 hours. After 24 hours the waste product was heated on a pan exposed to frequent stirring. When surface dry condition was reached the heater was turned off. Three samples were weighted and then dried in an oven at 105 °C until constant mass. The absorption was calculated by

$$A = \frac{m_{\mathrm{SD}} - m_{\mathrm{dry}}}{m_{\mathrm{dry}}} \tag{2.4}$$

In order to ensure the surface dry condition was archieved a metal cone mould and a tamper was used as described in DS/EN 1097-6:2013

The pycnometer method

The waste product was dried in an oven at 105 °C until constant mass. Approximately 150 g of waste product was added to three pycnometers. The dry weight was measured, and the pycnometers were filled halfway with airless water. The pycnometers were tilted 45 °and rolled back and forth in order to release air bubbles among the waste product. The pycnometers was then filled all the way with water, repeating the rolling back and

forth. The glass stopper was put in place and the outside of the pycnometers were dried with a paper towel on the outside. And the mass was weighted. This process was repeated at different time steps. Absorption was calculated by

$$A = \frac{m_{\text{pycno,t}} - m_{\text{pycno,t0}}}{m_{\text{dry}}} \tag{2.5}$$

2.11 Autogenous shrinkage using a dilatometer

Autogenous shrinkage was measured using the method described in ASTM C1698-09:2014. Two reference samples and two samples with the waste product was casted in a corrugated plastic mould, see Figure 2.4. The plastic moulds were closed with end plugs, upon which the measurement were performed. In order to ensure precision the moulds were marked so that they always was measured facing the same way.
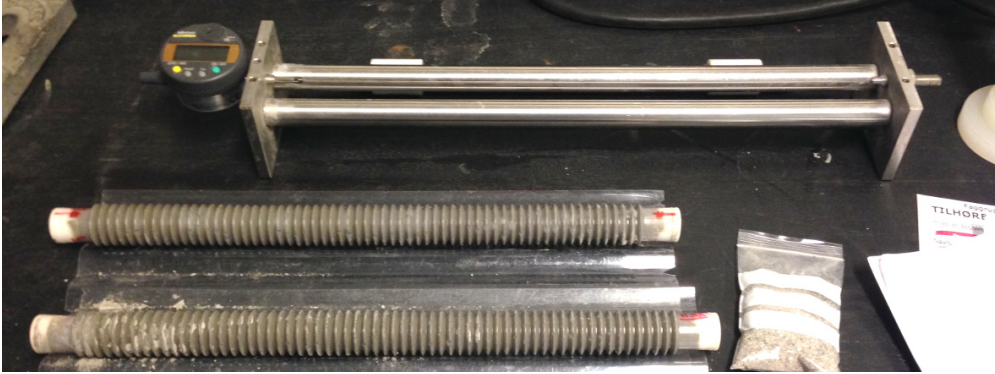
Figure 2.4: Autogenous shrinkage measurement setup. Front: two corrugated plastic moulds, back: The dilatometer. All measurements were performed relative to a 415 mm invar rod.

The length was measured relative to a 415 mm reference bar and the first measurement was performed 4 hours after casting. The autogenous strain in μm/m was calculated by using this formula from ASTM C1698-09:2014.

$$\epsilon_{\text{autogenous}} = \frac{R_t - R_{t0}}{L_{\text{ref}} + 2R_{t0} - 2L_{\text{plug}}} \cdot 10^6 \frac{\mu\text{m}}{\text{m}} \tag{2.6}$$

Where:

L_{ref}:	Length of the reference bar
R_{t0}:	Display measurement at time of final setting
R_t:	Display measurement at time = t.
L_{plug}:	Average length of plugs

3 Results and discussion

3.1 Survey of current customs

Eight danish concrete producing companies were asked about their use of high strength concrete and what they do to mitigate autogenous shrinkage. The letter is available in appendix A. Of the eight companies asked only four produced high strength concrete. One company mainly produced what is called ultra high performance concrete (UHPC) which because of its dense nature and low porosity does not have problems with autogenous shrinkage. Among the three other companies the solution were surface curing, cutting grooves in the concrete, casting smaller elements, using proper amount of reinforcement bars and fibres and accepting the shrinkage. None of the companies used internal curing agents or SAP. All the companies were asked about their definition of high strength concrete, most of the answers was concrete with higher strength than 50–60 MPa, one company regards 35–40 MPa as high strength and two companies answered that high strength concrete was concrete stronger than 100 MPa containing steel fibres. This underlines that no common definition is used when talking about high strength concrete.

3.2 Particle density and sand-mortar ratio

The particle density of the waste product was found to be $\rho_{\text{waste product}} = 2335 \pm 11\text{kg/m}^3$. Making the waste product a normal weight aggregate, as discussed in (Zou et al., 2015) this is helpful to avoid segregation based on too large differences in density. Using Equation 2.2 and table values from (Hansen et al., 2008) for the density of sand and mortar respectively, the sand-mortar ratio is estimated to lie within the interval $\alpha \in \{0.32 - 0.41\}$. Meaning more than half the waste product consists of mortar. It is preferable that this ratio is as low as possible since sand is not an effective agent for creating internal water reservoirs, having a low absorption on 2.7 % (Ghourchian et al., 2013).

3.3 Particle size distribution

Particle size distribution was performed on sand and on the waste product. Prior to laser diffractometry on the waste product, the waste product and sand was sieved through a sieve on 2 mm in net size — the maximum size for laser diffractometry and also the maximum size used in this project. This treatment were not enough for the waste product and the laser diffractometry failed because of too large particle sizes. The material was then sieved through a 1 mm sieve, the material held back and the material that passed the sieve was weighted. The waste product less than 1 mm was then used for laser diffractometry. Results was obtained, however the machine still experienced problems due to too large particle size. The reason for this was found to be the form of the waste product as can be seen in Figure 3.1 The elongated waste product particles
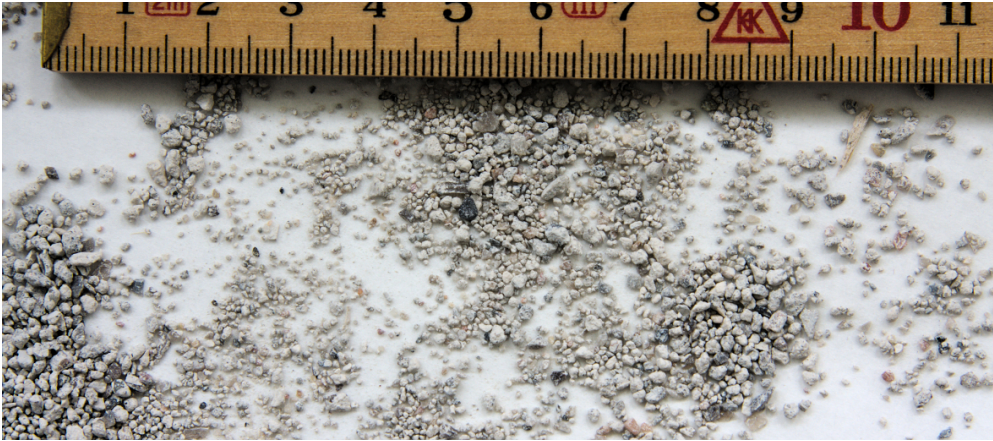

Figure 3.1: Photograph of the waste product.

could easily pass through a sieve on the short side, this makes the particle distribution harder to determine. Figure 3.2 contains the probability density function and cummulative density function for the sand and waste product size distribution. The curves as measured from laser diffractometry is shown in dashed line, where solid line indicates the curves that have been modified with data measured on the sieves. For the sand this has been done by adding the amount held back on the 2 mm sieve with maximum size of 4 mm. This has been added uniformly, since no data on the distribution in this interval was obtainable. For the waste product laser diffractometry still measured particles in the range of 1 to 2 mm although the waste product had been sieved through a 1 mm sieve. Almost 61 % of the waste product material was held back on the 1 mm sieve, this was uniformily added to the curve using the same arguments as for sand. Despite the elongated form affecting the results, the graph shows that the sand is more fine that the waste product. Since the waste product is replacing parts of the sand it would be desirable not to affect the particle size distribution to greatly. The elongated form of the particles are also an interesting result, since it is known that elongated aggregates have a tendency to decrease the workability of the fresh concrete (Mehta and Monteiro, 2014).
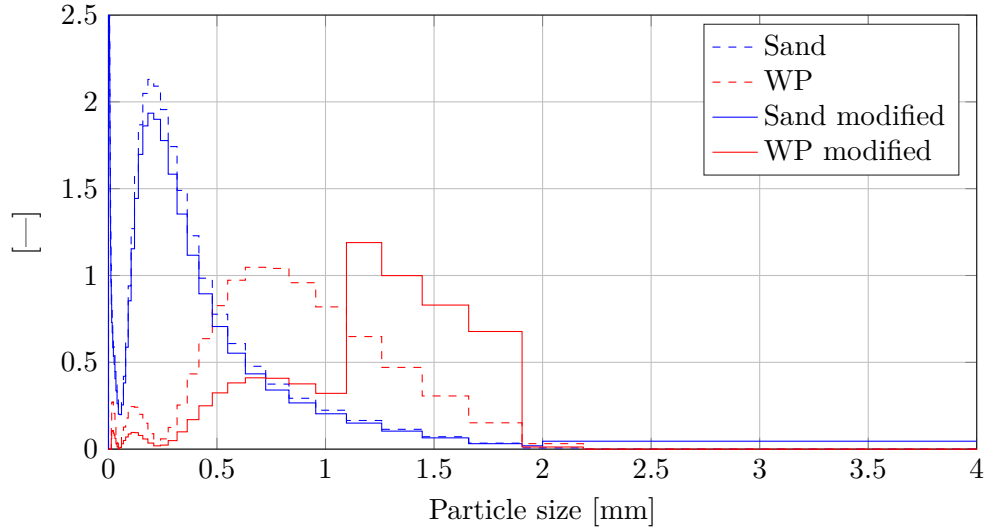
(a) PDF
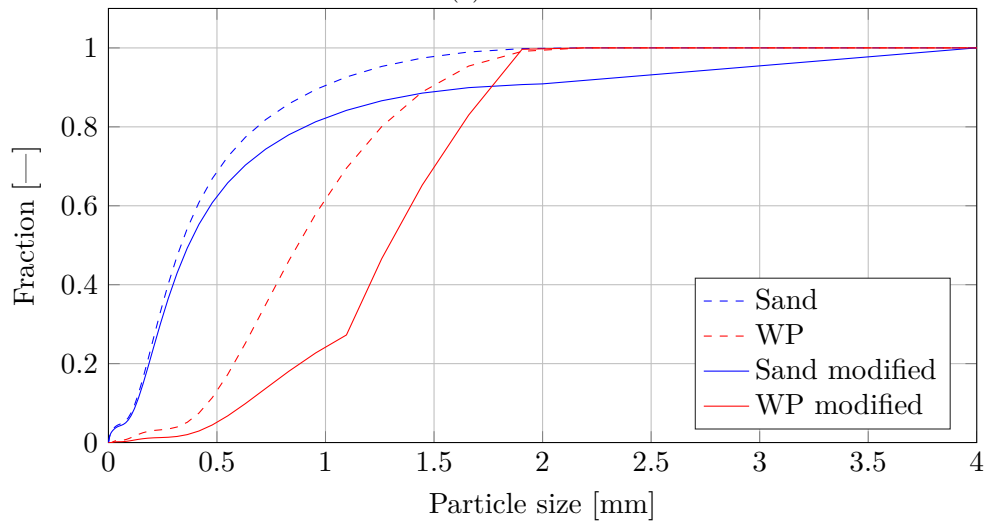
(b) CDF

Figure 3.2: Particle size distribution, probability density functions (PDF) and cummulative density function (CDF). The dashed line curves are based on the data from laser diffractometry alone, while the solid line represents data that has been modified with the weight measurement on sieves (2–4 mm for sand and 1–2 mm for the waste product.)

The workability is already a challenge when dealing with high strength concrete, so adding extra elongated particles is not desirable.

3.4 Loss on ignition (LOI)

During preparation of the waste product other materials was noticed. Mostly wood and plastics. Loss on Ignition was performed in order to quantify the amount of organic matter in the waste product. The loss on ignition was determined to be

$$LOI = 3.0 \pm 0.2\,\% \tag{3.1}$$

this is lower than the 5 % standard requirement in ASTM C1761/C1761M:2015. So the contamination with organic material should be less of a problem.

3.5 Uniaxial Compressive strength

Compressive strength was measured on reference mortar and mortar with 12 %, 22 % and 32 % WP replacement respectively. The results are available in Figure 3.3 with error bars showing one standard deviation. A statistical Smith-Satterthwaite test has been
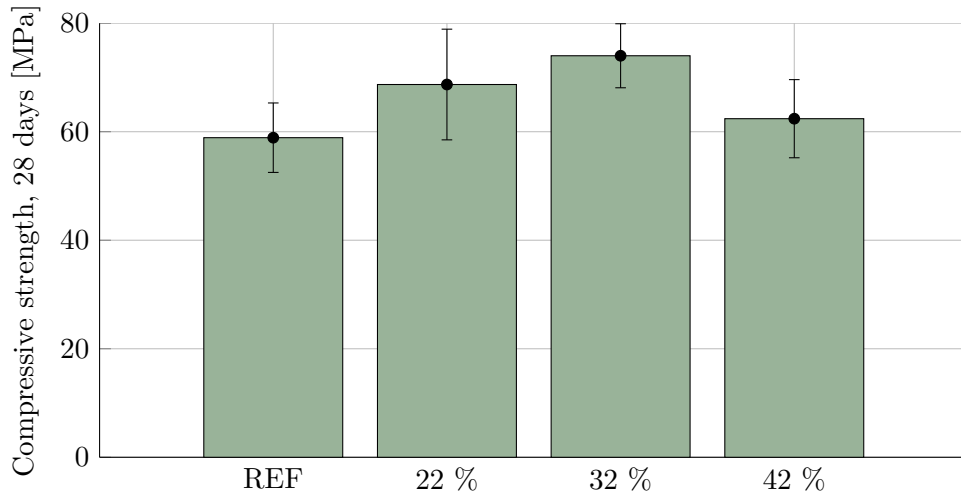
Figure 3.3: Compressive strength measured on $5 \times 5 \times 5$ cm cubic mortar samples at age 28 days.

carried out in order to check whether the measured strengths are significant different. The samples are with 95 % confidence not significantly different from eachother. See Table 3.1 for an overview of the statistical check and Appendix B. This is expectable since the addition of internal water reservoirs are not supposed to change the strength.

Table 3.1: Smith-Satterthwaite test value, t' for strength test specimens. If $|t'| > 4.303$ the samples are with 95 % confidence not equal eachother.

	REF	12	22	32
REF	—	$t' = -1.4$	$t' = -3.0$	$t' = -0.6$
12	—	—	$t' = -0.8$	$t' = 0.9$
22	—	—	—	$t' = 2.2$
32	—	—	—	—

3.6 Colour propagation test

The waste product was soaked in red coloured water and mortar samples were casted. Pictures of the mortar samples when opened after 14 days is available in Figure 3.4. No
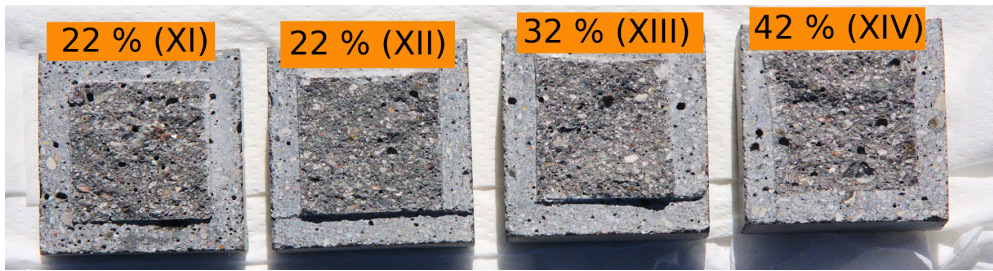
Figure 3.4: Cross sections of 14 days old mortar samples with different amount of added waste product soaked in colour water. The percentage indicates the amount of waste product added.

colour propagation is visible. Looking carefully red particles can be seen, however no colouring of the area around. This could mean that either no water has been released from the waste product particles or that the colour has remained in the waste product particles.

3.7 Desorption

Figure 3.5 and Figure 3.7 contains the desorption curves for the waste product, the equilibrium profile and as a function of time, respectively. Equilibrium profiles for LECA and Zeolite from (Ghourchian et al., 2013) has been included in Figure 3.5 for comparison. Ghourchian et al. (2013) concludes that LECA has a desorption curve that is suited for internal curing, while zeolite, in contrast, does not. The waste product investigated in this project lies somewhere in between. It does not release all its water at the highest relative humidities (>95 %) however it does release at least half of its water. How it fits in between the two materials from litterature might even be more clearly understood in Figure 3.6. ASTM C1761/C1761M:2015 requires that lightweight

Figure 3.5: Desorption of the waste product (0.5 mm – 2.0 mm). Plotted as a function of RH% after equillibrium is established.
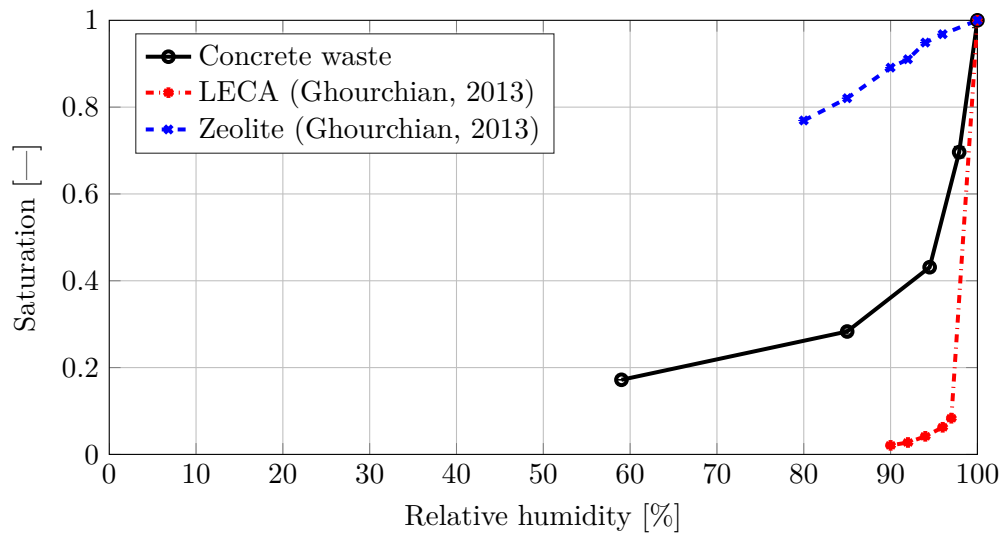
Figure 3.6: Normalized desorption of the waste product (0.5 mm – 2.0 mm). Plotted as a function of RH% after equillibrium is established.

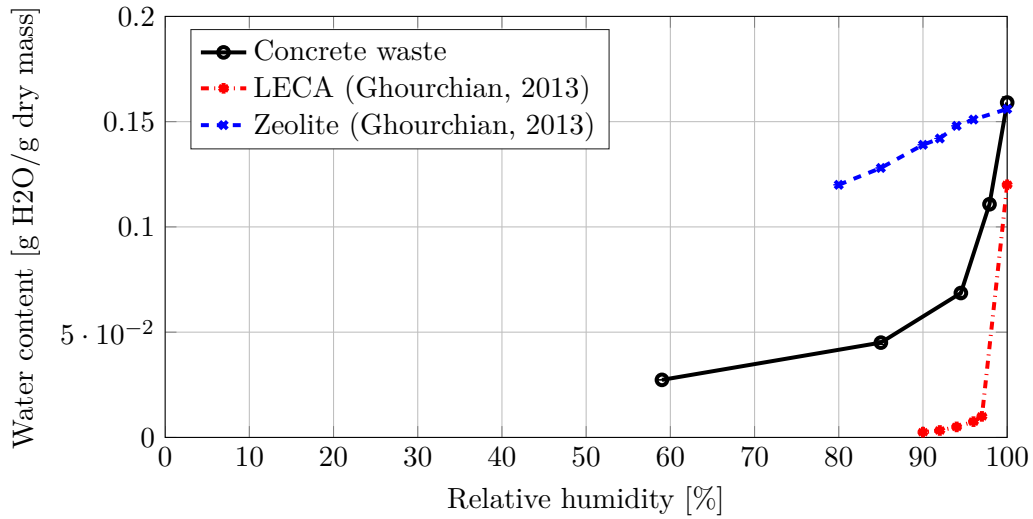aggregate for internal curing should release at least 85 % of the absorbed water when soaked for 72 hours and stored at 23 ± 1 °C in a relative humidity af 94 %. Figure 3.7 contains the desorption curve as a function of time. Following the curve for 94.5 % it is evident that the material does not come close to the standard requirement and only releases around 65 %. The desorption curve does not seem suited for internal curing. It should be noted though that this desorption curve is based on material soaked for 24 hours and not 72 hours as in the standard. However looking at the results from the pycnometer measurements of absorption in Figure 3.8, the material does not seem to soak more after 24 hours. Another remark to make is that the samples in 100 %RH seems to increase in weight, this is due to condensation on the sample containers.
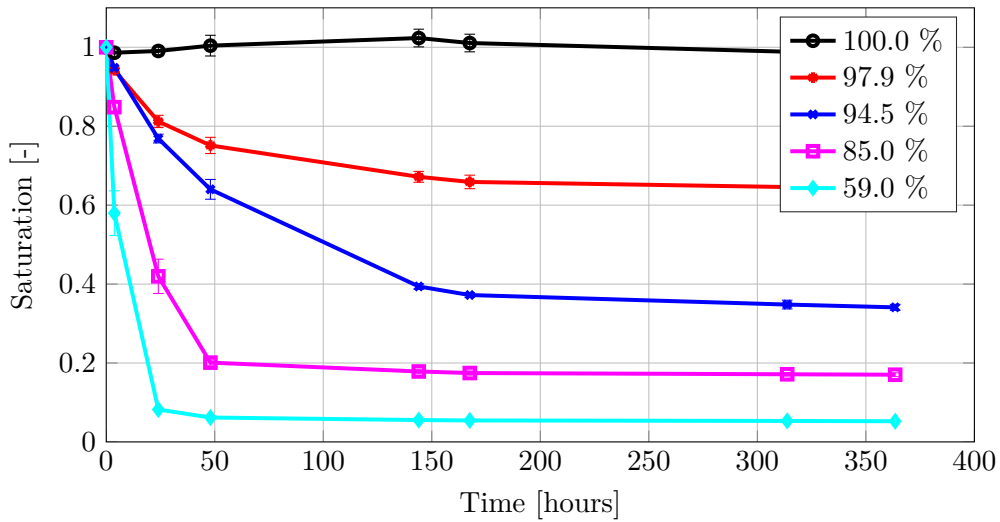
Figure 3.7: Normalized desorption of the waste product (0.5 mm – 2.0 mm). Plotted as a function of time

3.8 Absorption

Absorption determined by the pan method was determined to be $A_{24h,\text{WP,pan}} = 7.79 \pm 0.05$ % after soaking in distilled water for 24 hours. In contrast the absorption determined using the pycnometer method was $A_{24h,\text{WP,pycno}} = 1.68 \pm 0.18$ %. Figure 3.8 contains a plot of the absorption measured using the pycnometer method over time. It is clear that most absorption happens during the first 5 hours. After 24 hours the absorption does not increase anymore.

The disagreement between the two methods is peculiar and might arise from different limitations of the methods.

In *the pan method* the waste product was soaked in water for 24 hours and was then stirred on a pan until surface dry condition was archieved. The Archilles heel of this
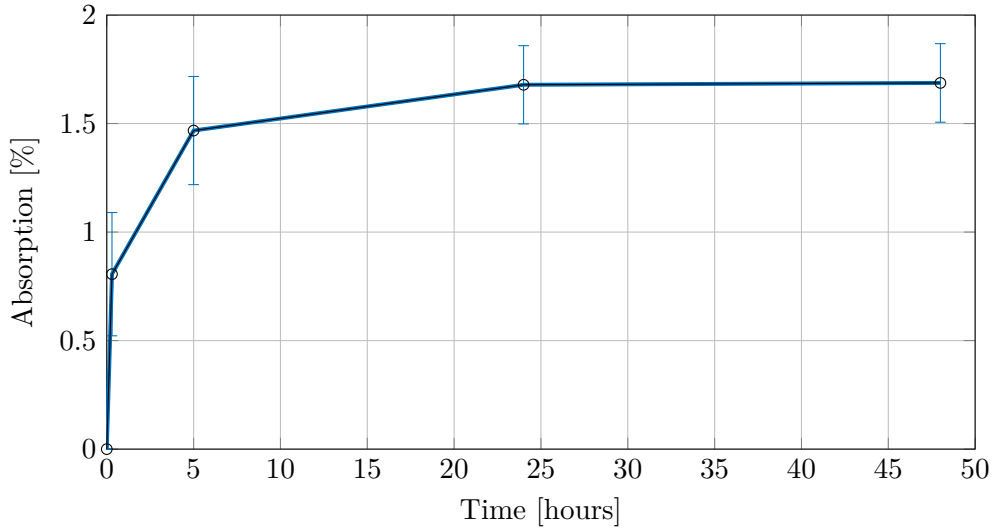
Figure 3.8: Absorption measured with the pycnometer method

method is in the way that surface dry condition is archieved and checked. When the material was tested for surface dry condition, using the metal cone mould, and it turned out positive, the heating was stopped. There is however a lag between when the heater is turned off and when the vapouration stops. Furthermore the pan does not heat the waste product uniformly, thereby demanding frequent stirring. Both effects makes the method more depended on the skill and choices of the experimenter, and it seems to make a rougher estimate of the absorption than say the pycnometer method.

The pycnometer method The pycnometer method does seem to be a more precise method than the pan method and less dependent on the experimenter. If prober care is taken to remove air bubbles, the method is quite simple to perform. The pycnometer method was carried out twice. First time around air bubbles was not removed, and the experiment was stopped after 48 hours when the experimenter noticed air bubbles in the waste product, see Figure 3.9. The second time care was taken to remove air bubbles, by rolling the pycnometers back and forth in a 45 °angle and it is the results from this experiment that is presented here. There was another difference between the two experiments. When the pycnometers was emptied after the first experiment, the waste product was quite easy to remove and did not seem to adhere to itself. After the second experiment, when the pycnometers were emptied after 72 hours the waste product seemed to adhere to itself and had to be removed by using a brush. It was not a strong adhesion, but it seems that some unreacted cement is reacting with the water. The effect of this adhesion could be that water is less freely to roam among the waste product particles. Resulting in a lower measured absorption. Due to time restraints a third experiment has not been carried out, however one could imagine that the effect of adhesion might be overcome by laying the pycnometers on there side (using a suitable stopper) and rolling them back and forth perhabs every hour or half hour.

Taking these considerations into effect both methods seems to have the possibility of
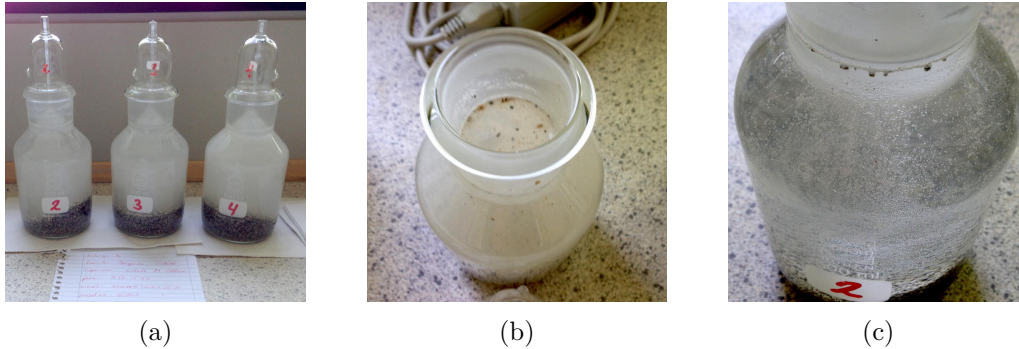
Figure 3.9: Absorption experiment using the pycnometer method. a) Three pycnometers with waste product and water, photo taken shortly after test start. b) View down one pycnometer without the stopper. Notice the lighter materials floating in the top and the muddy looking water. c) Air bubbles in pycnometer that has been left for 24 hours without stirring.

being off the true value. The estimate made by the pycnometer method is probably too low while the pan method might be a little high or not. It is hard to tell, but one plausible estimate would be to say that the true absorption value lies a place in between the ones measured by these two methods. Comparing with results from litterature on LECA $A_{24h,\text{LECA,litterature}} = 16.5 \pm 7.6$ ((Ghourchian et al., 2013), (Bentur et al., 2001), (Castro et al., 2011)) this is a low amount of absorption. And in order to mitigate autogenous shrinkage a higher value would be desirable. A lower absorption means that more material have to be used in order to mitigate autogenous shrinkage. Castro et al. (2011) comments that the absorption tends to decrease with fineness when particles are crushed. Using larger particle might be a solution if that is true for this material as well. This should be done with consideration of the fineness of the material replaced.

3.9 Autogenous shrinkage measured using a dilatometer

The autogenous shrinkage was measured using a dilatometer on samples with 22 % sand replaced by the waste product and compared to reference samples. Figure 3.10 shows the length change relative to length 4 hours after casting. The length of sample REF1 and WP2 decreases with time as would be expected of concrete. However for some unknown reason REF2 and WP1 increased in length during the first 24 and 48 hours respectively. Had the samples been compressed in the early stages due to improper handling, that might explain the increase between the first measurement and the 24 hours measurement. However it hardly explain the increase seen between 24 hours and 48 hours for WP1. Beside the samples were casted in the order REF1, REF2, WP1 and WP2 and was handled in that way. REF1 and REF2 were handled after 3 hours, where they were still to soft. Therefore 4 hours was chosen. It is therefore peculiar

that WP1 should have been affected when REF1 has not. The only internal effect that could explain an expansion known to the author is temperature. However the samples were to small for high temperatures developing, beside that would be common for all four samples. Some unknown external factor must be the cause. Instead of using the
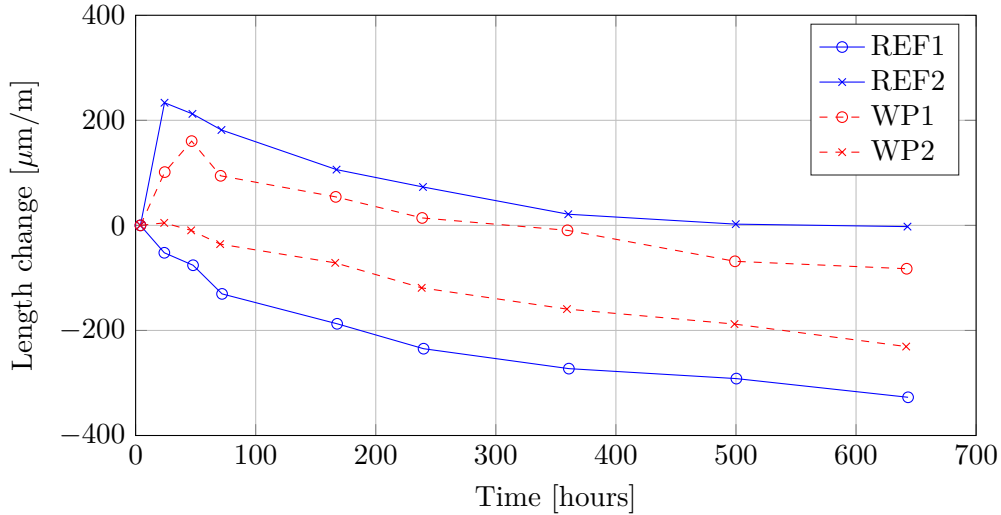
Figure 3.10: Length change of concrete samples, relative to length 4 hours after casting

4 hour measurement as the baseline Figure 3.11 uses the 48 hours measurement as the base. Both samples with waste product stays above the reference samples until the end of the experiment. However this seems to be mostly due to less shrinkage during the first 2–7 days. After this the rate of change seems to be approximately the same. At the end of the experiment after 27 days there is no difference between the samples. So based on these direct measurements the waste product does not seem to be effective as an internal curing agent. One remark though is that when the recipes for casting were designed the absorption value used was the one from the pan method, maybe replacing a larger portion of the sand could show an effect in future work.

3.10   Further research

Further research on the use of concrete waste as an internal curing agent could focus on confirming or contradicting the findings in this study using other batches of concrete. Instead of using concrete waste of unknown origin, using concrete waste from construction where the original strength is known could be beneficial. Projects might focus on desorption and absorption measurement on a variety of concrete waste materials.
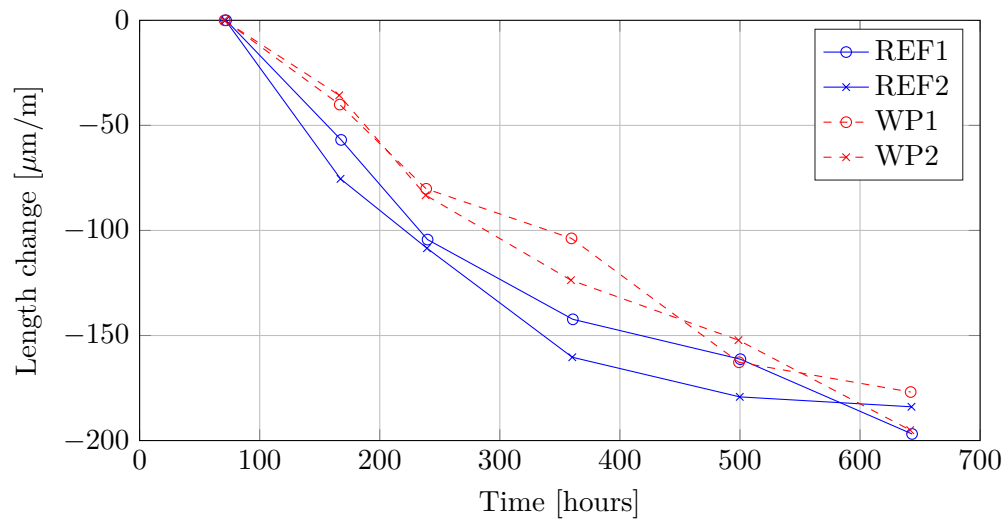
Figure 3.11: Length change of concrete samples, relative to length 48 hours after casting

4 Conclusion

Particle density was found to be $\rho_{\text{waste product}} = 2335 \pm 11\text{kg/m}^3$ resulting in an estimated sand-mortar ratio on $\alpha \in \{0.32 - 0.41\}$ and classifying the material as normal weight aggregate. Particle size distribution proved difficult to determine because of the elongated form of the waste product, however it did show that the waste product was more coarse than the sand. The elongated form of the waste product particles might decrease workability. Loss on ignition was found to be $3.0 \pm 0.2\,\%$ which lies within the ASTM C1761/C1761M:2015 requirement on 5 %. The strength did not change significantly with the addition of the waste product. Adding coloured water to the waste product showed no effect on the colour of the mortar when opened 14 days after casting.

The desorption curve of the waste product was not optimal for working as internal water reservoir and it did not seem to live up to the requirements in ASTM C1761/C1761M:2015. Disagreement was seen between the two methods used to determine the absorption value resulting in $A_{24h,\text{WP,pan}} = 7.79 \pm 0.05\,\%$ and $A_{24h,\text{WP,pycno}} = 1.68 \pm 0.18\,\%$ respectively, with the true value probably lying a place in between. Direct measurements of autogenous shrinkage did not show any effect of adding the waste product. However due to the low absorption, further research might find an effect by adding more waste product.

Overall there is not found any clear indications in this project that crushed concrete can be used as internal water reservoirs in high strength concrete in order to mitigate autogenous shrinkage. Further research could focus on other batches of the waste product and the effect of replacing more sand.

Acknowledgements

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Appendix

Appendix A

Letter to companies

Hi,

To whom this may concern.

I am a master student at the Technical University of Denmark and I am currently working on my master thesis. The project concerns reuse of crushed concrete waste as internal water reservoir in high strength concrete. Using the waste concrete as an internal curing agent in order to avoid/mitigate autogenous shrinkage caused by the low water-cement ratio.

I know that your time is valuable, however I hope that you want to answer 3–4 question regarding how you work with high strength concrete.

1. What do you define as high strength concrete?

2. How often do you produce high strength concrete compared to normal strength?

3. What do you do to mitigate autogenous shrinkage when you cast high strength concrete? (Internal curing with wet aggregate? Super Absorbant Polymeers? Do you and the customer accept the shrinkage?)

3.1 If you do use internal curing: Please describe your working process shortly. (I am especially interested in which material you use, how it is crushed and how you soak it in water)

If you wish, I would like to thank your company in the aknowledgements section of the report? Beside of this the answers will be handled anonymous. The answers will be used to get an overview of what is current working custom.

Thank you in advance

Kind regards
Leander Jørgensen
Phone no.: XX XX XX XX

Student at the Technical University of Denmark

Appendix B

Remarks on statistical analysis

Test of two mean values have been done in accordance with (Johnson, 2011). The test is carried out using a Smith-Satterthwaite test for comparing small samples with unequal standard deviations. The following assumptions have been made in order to do so:

- X, Y are random samples from two populations

- X and Y are independent

- Both populations are normal

The equation used to determine the test-value

$$t' = \frac{(\overline{X} - \overline{Y}) - \mu}{\sqrt{\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}}} \tag{B.1}$$

Where:
$\overline{X}, \overline{Y}$:	The mean of two random variable
S:	is the standard deviations
n:	Sample size

In order to use a t table, the degrees of freedom can be estimated by

$$\text{DOF}_{\text{est.}} = \left\lfloor \frac{\left(\frac{S_1^2}{n_1} + \frac{S_2^2}{n_2}\right)^2}{\frac{\left(S_1^2/n_1\right)^2}{n_1-1} + \frac{\left(S_2^2/n_2\right)^2}{n_2-1}} \right\rfloor \tag{B.2}$$

t-test values are found in tables in (Johnson, 2011).

Appendix C

Casting procedure

Mixing procedure

1. Cement in the mixing bowl

2. Add water:　　　　　*Time = zero*

　　　　　　　　　　　Low speed

　　　　　　　　　　　Mix for 30 sec.

3. Add SP, sand and/or waste concrete during the next 30 sec

4. Turn mixer on high speed – mix for additionally 30 sec

5. STOP for 90 sec

6. During the next 30 sec: scrape the mortar down from the rim of the bowl

7. Mix for additionally 60 sec at high speed.

Time = 240 sec　　=>　　Vibration table

Vibration table

1. Fill the form halway

2. Start the vibration – speed 45

　　　　　Count to 30 – stop

3. Fill the form all way

4. Start vibration – speed 45

　　　　　Count to 30, while scraping overtopping mortar

5. Cover sample with plastic

6. Belongs to …… - paper

Mixing procedure (Dilatometer)

1. Cement in the mixing bowl

2. Add water: *Time = zero*

                Low speed

                Mix for 30 sec.

3. Add SP, sand and/or waste concrete during the next 30 sec

4. Turn mixer on high speed – mix for additionally 30 sec

5. STOP for 90 sec

6. During the next 30 sec: scrape the mortar down from the rim of the bowl

7. Mix for additionally 60 sec at high speed.

Time = 240 sec   =>   Vibration table

Vibration table

1. Measure the length of each plug, place first plug in one end

2. Fill the form ¼ and tamp 25 times with the tamping rod.

3. Start the vibration – speed 55

        Count to 60 – stop

4. Fill the form 1/2 way, repeat tamping 25 times

5. Start vibration – speed 55

        Count to 60

6. Repeat for ¾

7. Fill form but leave space for the end plug, use tamper 25 times

8. Start vibration – speed 55

        Count to 60

8. Close the form with the last plug, handle the sample carefully

6. Belongs to …… - paper

Appendix D

Casting logbook

Sheet1

Sample	Cast date	Demould date	Type	End use	Recipeé Cement [g]	Water [g]	Sand [g]	WP [g]	SP [g]	w/c-ratio [-]	WP water content [%]	Comments
I	--	--	REF	TEST	267	95	700	0	4	0.36		Tendency to bleed?
II	--	--	REF	TEST	267	90	700	0	2	0.34		
III	15/03/16 14:00	16/3/16 14:00	REF	28d compr.	333	112	748	0	2.5	0.34		Dry, bad workability
IV	15/03/16 14:30	16/3/16 14:20	REF	28d compr.	349	118	719	0	3.5	0.34		
V	15/03/16 14:50	16/3/16 14:40	WP	28d compr.	349	118	564	155	3.5	0.34		More liquid than IV
VI	15/03/16 15:20	16/03/16 15:00	WP	28d compr.	349	118	492	227	3.5	0.34		More liquid than V
VII	30/03/16 14:35	31/03/16 14:30	REF	28d compr.	349	118	719	0	3.5	0.34	6.9	
VIII	30/03/16 14:50	31/03/16 14:35	WP	28d compr.	349	118	564	155	3.5	0.34	6.9	
IX	30/03/16 15:10	31/03/16 14:40	WP	28d compr.	349	118	492	227	3.5	0.34	6.9	
X	30/03/16 15:30	31/03/16 14:45	WP	28d compr.	349	118	420	299	3.5	0.34	6.9	
XI	06/04/16 13:50	07/04/16 13:50	WP	Colour	349	118	564	155	3.5	0.34	6.7	Dryer than last week
XII	06/04/16 14:20	07/04/16 14:20	WP	Colour	349	122	564	166	3.7	0.35	6.7	+4g water
XIII	06/04/16 14:40	07/04/16 14:40	WP	Colour	349	122	492	243	3.7	0.35	6.7	+4g water
XIV	06/04/16 14:55	07/04/16 14:55	WP	Colour	349	122	420	320	3.5	0.35	6.7	+4g water
XV	03/05/16 14:10	--	REF	autog.	403	139	618	0	2.4	0.34	12.3	
XVI	03/05/16 14:40	--	REF	autog.	403	139	618	0	2.4	0.34	12.3	
XVII	03/05/16 15:20	--	REF	autog.	403	139	351	286	2.4	0.34	12.3	
XVII	03/05/16 15:45	--	REF	autog.	403	139	351	286	2.4	0.34	12.3	

Samples I – VI has not been used for testing.

Appendix E

Particle density - measurements

Dato	
Navn	
Projekt	
Prøvemateriale	

Metode:
Efter Laboratoriehåndbogen, dgf-bulletin 15 (dgf15)
Efter DS/CEN ISO/TS 17892-3

Resultat
Beregning
Input

Bestemmelse af kornrumvægt
Sand

				1	2	3
Fra kalibrering af pyknometer						
Pyknometer nummer				8	2	1
Pykn. + prop (tomt)		m_0	g	48.9791	43.4429	47.5231
Pykn. + prop (vandfyldt)	W_2	m_1	g	150.7888	142.5256	149.2449
Temperatur ved kalibrering	T_k	T_1	°C	22	22	22
Densitet af vand ved T_k *	$\rho_{w,k}$	$\rho_{w;1}$	g/cm³	0.9978	0.9978	0.9978
Måling						
Pykn.+ prop + jord		m_2	g	51.1189	45.5838	49.7764
Pykn.+ prop + jord + vand	W_1	m_3	g	151.9864	143.738	150.5078
Temperatur	T	T_3	°C	23.5	23.5	23.5
Densitet af vand ved T *	$\rho_{w,t}$	$\rho_{w;3}$	g/cm³	0.99757	0.99757	0.99757
Jord - masse	W_s	m_4	g	2.1398	2.1409	2.2533
Jord - volumen	V_s		cm³	0.9209701	0.90786685	0.96930782
Korndensitet	ρ_s	ρ_s	g/cm³	2.32341963	2.35816519	2.32464853
Resultat - middel	ρ_s	ρ_s	g/cm³	**2.3354**		
Betegnelser fra	dgf15	DS				

* Se faneblad med vands densitet

Dgf-bulletin 15:

$$\rho_s = \frac{W_s}{V_s} = \frac{W_3 - W_0}{\dfrac{W_2 - W_0}{\rho_{w,k}} - \dfrac{W_1 - W_3}{\rho_{w,t}}}$$

DS/CEN ISO/TS 17892-3:

$$\rho_s = \frac{m_4}{\dfrac{m_1 - m_0}{\rho_{w;1}} - \dfrac{m_3 - m_2}{\rho_{w;3}}}$$

Der bør ikke være stor forskel på temperaturen ved kalibrering og måling.
Der kan evt. foretages kalibrering ved flere temperaturer, eller udføres en teoretisk
korrektion af volumen af pyknometer:

$$V_{pyk;test} = \frac{W_2 - W_0}{\rho_{w,k}} \alpha$$
$$\alpha = 1 + 3 \cdot 0.000003 \left(T - T_k \right)$$

$$V_{pyk;3} = \frac{m_1 - m_0}{\rho_{w;1}} \alpha$$
$$\alpha = 1 + 3 \cdot 0.000003 \left(T_1 - T_3 \right)$$

file:///home/leander/Dropbox/Danmark Tekniske Universitet/Master thesis/Lab tests/Particle density/160201_ko

Appendix F

Laser diffractometry

Laser Diffractometry on concrete

MASTERSIZER 2000

Result Analysis Report

Sample Name:
Averaged Result-Beton tør

SOP Name:
askeTør

Measured:
Wednesday, May 04, 2016 10:48:13 AM

Sample Source & type:
Paris

Measured by:
malm

Analysed:
Wednesday, May 04, 2016 10:48:15 AM

Sample bulk lot ref:
123-ABC

Result Source:
Averaged

Particle Name:
aske

Accessory Name:
Scirocco 2000

Analysis model:
General purpose

Sensitivity:
Enhanced

Particle RI:
1.500

Absorption:
0

Size range:
0.020 to 2000.000 um

Obscuration:
0.61 %

Dispersant Name:

Dispersant RI:
1.000

Weighted Residual:
1.774 %

Result Emulation:
Off

Concentration:
0.0121 %Vol

Span :
1.169

Uniformity:
0.362

Result units:
Volume

Specific Surface Area:
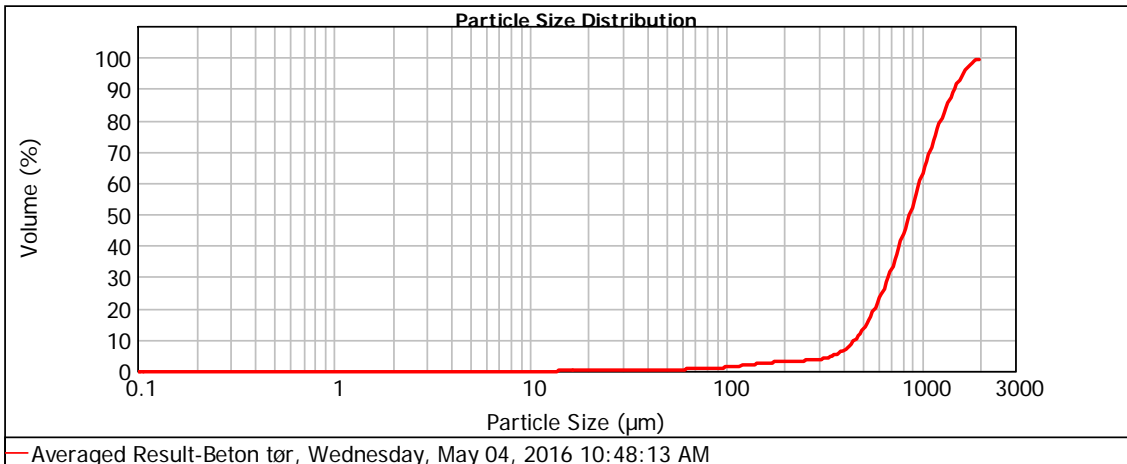0.00999 m²/g

Surface Weighted Mean D[3,2]:
600.660 um

Vol. Weighted Mean D[4,3]:
917.228 um

d(0.1): 459.046 um   **d(0.5):** 870.705 um   **d(0.9):** 1476.708 um

Averaged Result-Beton tør, Wednesday, May 04, 2016 10:48:13 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.00	11.482	0.00	120.226	0.43	1258.925	8.78
0.011	0.00	0.120	0.00	1.259	0.00	13.183	0.03	138.038	0.41	1445.440	6.56
0.013	0.00	0.138	0.00	1.445	0.00	15.136	0.06	158.489	0.35	1659.587	3.73
0.015	0.00	0.158	0.00	1.660	0.00	17.378	0.07	181.970	0.24	1905.461	0.89
0.017	0.00	0.182	0.00	1.905	0.00	19.953	0.08	208.930	0.15	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.00	22.909	0.08	239.883	0.21	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.00	26.303	0.08	275.423	0.51	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.00	30.200	0.07	316.228	1.19	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.00	34.674	0.04	363.078	2.33	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.00	39.811	0.02	416.869	3.93	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.00	45.709	0.00	478.630	5.86	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.00	52.481	0.02	549.541	7.92	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	0.00	60.256	0.07	630.957	9.79	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	0.00	69.183	0.13	724.436	11.17	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	0.00	79.433	0.21	831.764	11.81	8709.636	0.00
0.079	0.00	0.832	0.00	8.710	0.00	91.201	0.30	954.993	11.59	10000.000	0.00
0.091	0.00	0.955	0.00	10.000	0.00	104.713	0.38	1096.478	10.53		
0.105		1.096		11.482		120.226		1258.925			

Operator notes: *Average of 3 measurements from 160504Leander*

Malvern Instruments Ltd.
Malvern, UK
Tel := +[44] (0) 1684-892456 Fax +[44] (0) 1684-892789

Mastersizer 2000 Ver. 5.60
Serial Number : MAL1017217

File name: 160504Leander
Record Number: 15
5/4/2016 10:59:29 AM

Laser Diffractometry on sand

MASTERSIZER 2000

Result Analysis Report

Sample Name:
Sand - Average

Sample Source & type:
Paris

Sample bulk lot ref:
123-ABC

SOP Name:
Sten

Measured by:
malm

Result Source:
Averaged

Measured:
Wednesday, May 04, 2016 10:14:21 AM

Analysed:
Wednesday, May 04, 2016 10:14:22 AM

Particle Name:
Sten

Particle RI:
1.500

Dispersant Name:
Water

Accessory Name:
Hydro 2000SM (A)

Absorption:
0.1

Dispersant RI:
1.330

Analysis model:
General purpose

Size range:
0.020 to 2000.000 um

Weighted Residual:
0.594 %

Sensitivity:
Enhanced

Obscuration:
14.73 %

Result Emulation:
Off

Concentration:
0.1938 %Vol

Specific Surface Area:
0.08 m²/g

Span :
2.557

Surface Weighted Mean D[3,2]:
74.991 um

Uniformity:
0.771

Vol. Weighted Mean D[4,3]:
450.245 um

Result units:
Volume

d(0.1): 123.651 um   **d(0.5):** 333.877 um   **d(0.9):** 977.484 um

Particle Size Distribution

— Sand - Average, Wednesday, May 04, 2016 10:14:21 AM

Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010		0.105		1.096		11.482		120.226		1258.925	
0.011	0.00	0.120	0.00	1.259	0.05	13.183	0.18	138.038	2.83	1445.440	2.13
0.013	0.00	0.138	0.00	1.445	0.05	15.136	0.18	158.489	3.82	1659.587	1.53
0.015	0.00	0.158	0.00	1.660	0.06	17.378	0.18	181.970	4.81	1905.461	0.85
0.017	0.00	0.182	0.00	1.905	0.08	19.953	0.19	208.930	5.74	2187.762	0.20
0.020	0.00	0.209	0.00	2.188	0.09	22.909	0.19	239.883	6.47	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.11	26.303	0.20	275.423	6.95	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.13	30.200	0.21	316.228	6.95	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.14	34.674	0.20	363.078	7.11	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.16	39.811	0.19	416.869	6.98	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.17	45.709	0.17	478.630	6.61	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.18	52.481	0.15	549.541	6.08	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	0.19	60.256	0.17	630.957	5.51	6606.934	0.00
0.060	0.00	0.631	0.03	6.607	0.20	69.183	0.25	724.436	4.95	7585.776	0.00
0.069	0.00	0.724	0.05	7.586	0.20	79.433	0.43	831.764	4.46	8709.636	0.00
0.079	0.00	0.832	0.05	8.710	0.20	91.201	0.76	954.993	4.02	10000.000	0.00
0.091	0.00	0.955	0.05	10.000	0.19	104.713	1.27	1096.478	3.61		
0.105	0.00	1.096	0.05	11.482	0.19	120.226	1.97	1258.925	3.17		
									2.68		

Operator notes:

Appendix G

Loss on ignition (LOI) - measurements

Sheet1

LOI	1	2	3	AVG	SD
Weight: container	14.3191	10.0787	13.7200		
Container + concrete BEFORE	18.0343	14.321	18.4401		
Container + concrete AFTER	17.9304	14.1832	18.2994		
LOI [g]	0.1039	0.1378	0.1407		
LOI [-]	0.0280	0.0325	0.0298	0.03	0.00
LOI [%]	2.7966	3.2482	2.9809	3.01	0.23

All measurements in gram

Appendix H

Compressive strength - measurements

Sheet1

Sample	Type	KN*	kN	mm	Mpa	
VII A	REF		157	161	0.8	62.8
VII B	REF		156	160	0.8	62.4
VII C	REF		129	133	0.6	51.6
VIII A	12 pr.		198	202	0.7	79.2
VIII B	12 pr.		147	151	0.7	58.8
VIII C	12 pr.		170	174	0.7	68
IX A	22 pr		193	197	0.7	77.2
IX B	22 pr		168	172	0.7	67.2
IX C	22 pr		194	198	0.7	77.6
X A	32 pr		141	145	0.8	56.4
X B	32 pr		151	155	0.6	60.4
X C	32 pr		176	180	0.7	70.4

Appendix I

Desorption - measurements

Sheet1

Table 1: Measurements

	kontrol	dk	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Weight of container																	
			34.72635	34.47705	36.00840	35.30331	37.18759	36.61518	37.52440	35.60134	34.18621	35.46347	34.08192	33.12817	33.81635	37.68519	32.27535
13/04/16 11:15	8.65447		45.03667	44.41992	49.78339	45.57146	51.71374	50.21931	52.63314	50.43818	49.88805	47.92600	43.73401	45.73831	46.90565	53.94454	43.79214
13/04/16 15:00	8.65447	0.00000	45.03490	44.40107	49.74279	45.49468	51.62032	50.11746	52.53713	50.33926	49.79164	47.71118	43.52993	45.51013	46.22339	53.22583	43.12256
14/04/16 11:15	8.65454	0.00007	45.03461	44.41610	49.74490	45.32365	51.41026	49.89361	52.19448	50.00062	49.47025	47.08557	42.96763	44.86578	45.43455	52.12155	42.49383
15/04/16 11:15	8.65449	0.00002	45.01790	44.41196	49.84030	45.24299	51.31176	49.78855	52.00476	49.73382	49.20674	46.71285	42.76790	44.48034	45.40282	52.07608	42.46750
19/04/16	8.65451	0.00004	45.05507	44.42823	49.86519	45.15189	51.15586	49.65793	51.51412	49.33344	48.71815	46.68342	42.73404	44.44866	45.39333	52.06316	42.45822
20/04/16 10:45	8.65436	-0.00011	45.04267	44.41023	49.84258	45.12493	51.13719	49.64609	51.47247	49.28542	48.68617	46.67596	42.72934	44.44338	45.39151	52.06112	42.45651
26/04/16 13:00	8.65459	0.00012	45.00322	44.38305	49.82268	45.11803	51.08420	49.64053	51.43841	49.23203	48.63931	46.67046	42.72499	44.44070	45.38927	52.05962	42.45470
28/04/16 14:45	8.65449	0.00002	45.00823	44.37951	49.80966	45.10440	51.07907	49.62812	51.41657	49.22286	48.63044	46.66964	42.72266	44.43886	45.38806	52.05774	42.45370
Dry measurements																	
10/05/16 13:40	8.65449	0.00002	43.60563	43.03548	47.88355	44.14579	49.67728	48.32145	50.50263	48.35989	47.71454	46.18899	42.34272	43.95878	45.07906	51.67723	42.18244
11/05/16	8.65447	0.00000	43.60526	43.03480	47.88301	44.14488	49.67612	48.32189	50.50160	48.35926	47.71328	46.18954	42.34134	43.95878	45.07854	51.67732	42.18156
Dry measurements Processed																	
650			8.87926	8.55841	11.87513	8.84246	12.48967	11.70625	12.97821	12.75853	13.52831	10.72550	8.26078	10.83059	11.26269	13.99202	9.90707
			8.87891	8.55775	11.87461	8.84157	12.48853	11.70671	12.97720	12.75792	13.52707	10.72607	8.25942	10.83061	11.26219	13.99213	9.90621
		GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD

Appendix J

Absorption - measurements

Sheet1

Absorption – pan method

Vægt af glas

	A1	A2	A3	A1*	A2*	A3*	dA1	dA2	dA3	dA1 %	dA2 %	dA3 %	AVG d %	STD d %
	108.42	110.38	107.94											
16/02/2016 16:00	256.35	254.81	257.61	147.93	144.43	149.67								
17/02/2016 15:10	245.66	244.31	246.85	137.24	133.93	138.91	10.69	10.5	10.76	7.79	7.84	7.75	7.79	0.05

Absorption – pycnometer method

	Dato	t (h)	20 før	20	Abs	Dato	t (h)	30 før	30	abs	t (h)	40 før	40	abs
Pycnometer + stopper	-			365.14					363.19				363.67	
Pycno + dry WP	2016/05/30 10:00			519.31					519.09				513.18	
Pycno + WP + H2O	2016/05/30 10:10	0		1037.62		2016/05/30 10:19	0		1035.65		0		1032.62	
	2016/05/30 10:37	0.45	1036.68	1038.69	0.69	2016/05/30 10:42	0.38	1034.64	1037.41	1.13	0.4	1032.11	1033.51	0.60
	2016/05/30 15:10	5	1037.95	1039.44	1.18	2016/05/30 15:12	4.88	1036.75	1038.14	1.60	4.9	1033.67	1035.05	1.63
	2016/05/31 10:05	23.9	1038.2	1039.89	1.47	2016/05/31 10:13	23.9		1038.39	1.76	23.9		1035.32	1.81
	2016/06/01 10:20	48.2	1038.57	1039.9	1.48	2016/06/01 10:25	48.1	1037.17	1038.42	1.78	48.1	1033.88	1035.32	1.81
	2016/06/02 10:32		1038.16	1039.74	1.38	2016/06/02 10:34		1036.86	1038.43	1.78		1033.49	1035.33	1.81

Additional date column (sample 40):

Dato
2016/05/30 10:22
2016/05/30 10:45
2016/05/30 15:16
2016/05/31 10:16
2016/06/01 10:26
2016/06/02 10:36

Appendix K

Autogenous shrinkage - Dilatometer measurements

Sheet1

Autogenous shrinkage measurements

XV Date time	length [mm]	XVI Date time	length [mm]	XVII Date time	length [mm]	XVIII Date time	length [mm]
03/05/16 14:10	--	03/05/16 14:40	--	03/05/16 15:20	--	03/05/16 15:45	--
03/05/16 18:10	10.302	03/05/16 18:40	12.47	03/05/16 19:20	12.642	03/05/16 19:40	9.202
04/05/16 14:05	10.28	04/05/16 14:45	12.569	04/05/16 15:35	12.685	04/05/16 15:35	9.204
05/06/16 14:00	10.27	05/06/16 14:00	12.56	05/06/16 14:00	12.71	05/06/16 14:00	9.198
06/05/16 14:10	10.247	06/05/16 14:10	12.547	06/05/16 14:10	12.682	06/05/16 14:10	9.187
10/05/16 14:00	10.223	10/05/16 14:00	12.515	10/05/16 14:00	12.665	10/05/16 14:00	9.172
13/05/16 14:00	10.203	13/05/16 14:00	12.501	13/05/16 14:00	12.648	13/05/16 14:00	9.152
18/05/16 15:00	10.187	18/05/16 15:00	12.479	18/05/16 15:00	12.638	18/05/16 15:00	9.135
24/05/16 10:30	10.179	24/05/16 10:30	12.471	24/05/16 10:30	12.613	24/05/16 10:30	9.123
30/05/16 09:30	10.164	30/05/16 09:30	12.469	30/05/16 09:30	12.607	30/05/16 09:30	9.105