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The effect of using aplite as a cementitious material

Tensile strength and particle size reduction

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ABSTRACT

The compressive strength of cement cubes containing aplite with different grain size distributions was measured. Varying degrees of milling and particle size reduction was looked upon. The aplite containing cubes were then compared with test cubes containing fly ash and neat cement. Density and particle size distribution was also measured.

The results showed that, the greater the amount of milling the aplite, the higher the measured compressive strength was.

INTRODUCTION

The earth faces many environmental challenges, but the emission reduction of carbon dioxide is probably the most important one. Cement production stands for 5 % of the human made CO₂ emission. Traditional cement production gives climate emission from two sources: It demands intense heat and thereby much energy to heat up the kilns that burns the raw materials. The raw materials give off CO₂ when they burn. This process is known as calcination, where carbon dioxide is liberated from calcium carbonate to form calcium oxide [1].

Use of substitute materials mixed in cement or as a component in the concrete recipes, is the most important agent to reduce the emission of carbon dioxide in a global relation. Pozzolanas are materials that mainly contain reactive silica and/ or alumina (not less than 70 %). On their own they have little or no binding property, but when mixed with lime in the presence of

water, they will set and harden like cement. Pozzolanas are an important ingredient in the production of alternative cementing material to Portland cement. When used in combination with cement they have a lot of advantages, such as long-term strength at high temperatures, resistance to sulphate attack, improved workability and they reduce costs considerably [2].

The petroleum industry wants to use the aplite concrete in well cementing. It may solve a lot of problems the traditional cement causes. The most important is that it does not shrink during hardening (hydration) and that it endures high temperatures. The cement must be stress/compression- and temperature resistant under more and more demanding and complex well conditions [3].

Conclusion from a Bachelor thesis by a student at UiS has shown that aplite could have an effect over longer periods of time [4]. Milling cement and pozzolana together could have a positive effect on the compression strength and durability. Other researching shows that, the greater the amount of milling, the finer the pozzolana will become and the rate of reaction will increase. The pozzolana and cement must be mixed as thoroughly as possible and is best achieved by use of a ball mill [2].

Same type of cement, fly ash, aplite and water/cement ratio (w/c) has been used in all test cubes. Only the grain size, milling time and the mixing are the differences in each concrete sample. By eliminating and decreasing the variables in

the cement mixture, the easier it would be to examine the differences.

Parts of the thesis were done in cooperation with Anders Nordhagen.

MATERIALS AND METHODS

Aplite

In this work we have used aplitite as a substitute material for cement. Aplitite (AP) is a natural pozzolan and usually white grey or pinkish in colour. It is a light and fine-grained intrusive rock, in which quartz and feldspar are the dominant minerals. The aplitite we have used comes from Norway, or more specific from Finnvollalen at Namsskogan. A large occurrence is found in this area [5]. The aplitite sample we used was very fine grained, most likely crushed and milled. The specification sheet for the aplitite used in this thesis is given in Fig. A1 in Appendix A. It shows a particle size distribution having a d10 of 6.58 μm , a d50 of 35.74 μm and d90 of 89.52 μm .



Figure 1: Aplitite in different sizes.

Fig. 1 shows aplitite in different particle sizes. The aplitite at the right hand side was the original aplitite used in the study. The measured grain sized distribution for this aplitite is illustrated in Fig. A2 in Appendix A.

Fly ash

For comparison we have used a cement containing fly ash. The fly ash (FA) which is used in this thesis is delivered by

Norcem AS. It is used to increase the matrix volume and reduce the cement consumption. Fly ash has relatively low water demand and can replace up to 40 % of cement in some concrete mixtures. Fly ash is one of the residues generated in the combustion of coal and waste. Depending upon the source and makeup of the coal being burned, the components of fly ash vary considerably. All fly ashes include substantial amounts of silicon dioxide (SiO_2) and calcium oxide (CaO) [6].

There are many good reasons to use fly ash as a substitute material in cement. The fly ash composition makes it react in the concrete. This results in a denser concrete, which makes it more resistant against decomposition. The fine particles give the concrete improved casting properties. Environmentally the fly ash gives lower energy consumption per ton cement, which results in reduced CO_2 emission. It also reduces the amount of waste in landfills [7].

A more accurate density measurement was done after the first castings. We used a density 1.9 g/cm^3 in our first mixture calculations. The new density measurements showed 2.3 g/cm^3 . Since we already had started the casting, 1.9 g/cm^3 was also used in later calculations. This was most appropriate with regard to later comparison. Measured particle size distribution is shown in Fig. A3 in Appendix A.

Industry cement

Norcem industry cement (IC) is custom-made cement well adapted to Norwegian winter climate. Blaine fineness is 510 m^2/kg , which is substantially finer than regular cement. Because of its fineness, the industry cement gives increased toughness in fresh concrete. The fresh concrete is more stable and the risk of separation is decreased. With such properties the cement makes it possible to carry out casting at winter, in a rational and

economic way. The rapid solidity development makes it well suited for use in production of concrete elements and other concrete products. High heat generation makes it unsuitable for large, massive constructions [8].

The cement mixtures we made were sent to Norcem for analyze. Analyzes showed that the industry cement contained CO₂. The cement was also pre hydrated, which means that water was present [9]. CO₂ in concrete reduces the compressive strength. This cement package was used in some of our cement mixtures. For the last mixtures a new cement package was used. Which cement package used in which test series, is given in Table C1 in Appendix C. The first industry cement was used in the particle size distribution measurements and is illustrated in Fig. A4 in Appendix A.

Density testing

Before starting the milling and composition of our concrete samples, the density of each material was measured. The density is normally informed by a document attached to the materials package. The fly ash density was not described in the document.

Density testing for aplite, fly ash and industry cement was conducted. Only the measured result of the fly ash density was used. Table 1 show the densities that were used in the sample preparations part.

Table 1: Material densities used in mixture calculations.

| Material | Density |
|-----------------|------------------------|
| Aplite | 2.65 g/cm ³ |
| Fly ash | 1.91 g/cm ³ |
| Industry cement | 3.12 g/cm ³ |

The procedure started with placing the samples in a 100°C heat closet for at least 12 hours. We weighted the samples before and after the heat treatment. In that way we were able to find the water content

in each sample. The water content in the samples was negligible. After having been cooled down to room temperature, the samples were placed in a volumetric flask (1000 ml). The flask alone and with the sample were placed on a weight and measured. Distilled water was added and filled up to the 1000 ml line. The volumetric flask, the material and the added distilled water were all measured on the weight. Some aplite density measurements are shown in Table B1 in Appendix B. With variable results we decided to use the densities described in the documents. A more accurate density measurement method could have been carried out and is described in reference [10].



Figure 2: Volumetric flask filled with aplite and water up to the 1000 ml line.

Sieving

Sieving of the materials was done to find the particle size distribution and to scalp the components to a requested size. To utilize this job, we used a 300 mm Impact Laboratory Test Sieve. Five different sieves, 125 µm, 71 µm, 63 µm, 40 µm and 20 µm were used.

Finding the particle distribution for each material was done by placing the samples and the sieves in a 100°C heat

closet during night. All the sieves were cleaned before and after each test. Each sample, sieve and rubber cube was weighted measured and noted. In this way it was easy to find the amount in each sieve at the end of the test. 3-4 rubber cubes were placed in each sieve to increase the efficiency of the sieving. The sieving was done automatically by the machine. Some of the material samples became lumpy. The lumps were crushed manually with a spoon. Weight measurements of the sieves were done each hour, to find out when the sieving was finished. All sieving was utilized with same amount of time. Measured particle size distribution can be found in Appendix A.

The scalping was done in almost the same way. Number of sieves used was reduced from five to two. The upper sieve had no practical use. Particles that passed the lower scalping sieve were gathered. The reminding particles were milled one more time in the ball mill.

To find the particle size distribution for milled and scalped aplite samples, a AcoustoSizer II developed by Colloidal Dynamics was utilized. Key numbers are described as d15, d50 and d85.

Milling

For mixing and pulverizing the materials, two different types of mills were used. To mix the materials lightly together, we used a Los Angeles mill. The mill was cleaned before and after each milling. The samples were milled for approximately 40 minutes (one round = one second).



Figure 3: Los Angeles Mill

To reduce the grain size of a mineral, a Retsch Planetary Ball Mill PM 100 was utilized. The machine is used wherever the highest degree of fineness is required. We used the PM 100 with one milling station with a 250 ml jar volume. 40-50 g of the materials was milled at the time. First the millings were done in 7 minutes. After some time of milling, the different modifications were tested. Since the milling and sieving was carried out parallel, the milling time was extended. Milling speed was set to 400 rpm. The milling operation was set to 5 minutes intervals with 2 minute breaks. Because of the high heat generation, the breaks were important. After the milling operation was done the particles were placed in the sieving machine. This operation was done continuously until the needed amount for our samples was reached. The milling jar contained 12 balls. The difference in speeds between the balls and milling jar produces an interaction between frictional and impact forces, which releases high dynamic energies. Interplay between the forces produces a high and effective degree of size reduction [11].



Figure 4: Retsch Planetary Ball Mill PM 100

Mixing machines

For mixing large quantum concrete, an Eibenstock Automix 1800 was used. It rotates at 70rpm. For less quantum concrete we used a Hobart Mixer.



Figure 5: Eibenstock Automix 1800

Compression testing machine

To execute the compression testing of the concrete samples, a Toni Tech machine was used. All cubes were pressed and registered with use of TestXpert software. The machine's upper load capacity was 3000kN.

EXPERIMENTAL CONDITIONS

Sample composition

As mentioned in the introduction the amount of variables were reduced. The easiest way was to choose a standard pozzolan/cement ratio. The ratio was set to 20/80 by volume. All concrete samples contained the same amount of water. We choose 0.38 as water/cement ratio by weight. Cement in this case, includes industry cement and a pozzolana. In this way it could be attached importance on the particle size, particle distribution and mixing.

By use of known and tested densities of the used materials, we could compose different cement mixtures. The different mixtures are described in Appendix B.

Sample preparation

Cube moulds were used when casting the concrete. The mould dimension was 50 x 50 x 50mm. With use of smaller moulds than normal, the needed cement amount was reduced. The moulds were

correctly assembled and lubricated with Vaseline before casting. Before starting the mixing and casting, it was important to find the right amount of cement and pozzolana for each sample. By deciding the amount of cubes needed, the volume was calculated. The mixtures and amount of cubes made per mixture are described in Appendix B. Conversion from volume to weight was done by multiplying known volumes with the densities listed in Table 1.

$$M = V \cdot \rho \quad (1)$$

M is mass of the material, V is volume and ρ is the density of the material.

All concrete cubes were made in the same way. The mixing of the concrete was done by mixing the dry materials lightly in the mixer. If the materials were milled together already, this was not necessary. After that the water was measured and added into the dry mix. The concrete mixture was mixed in approximately three minutes till a homogeneous structure was accomplished. The concrete slurry was then filled in the casting moulds 1/3 at the time. An iron rod was thrused in and out 25 times the first layer. This was also done in the next layer. The thrusting was done to attach the layers and to even out the concrete. A trowel was used to even out surface [12]. At the end the moulds were covered with plastic foil to prevent dehydration. The samples were stored at room temperature for 24 hours. The moulds were then dismantled and the cubes were marked with a number. To prevent cracks and dehydrated cubes, the test samples were placed in a curing bath as soon as possible after casting.

The tests which were carried out were divided into different groups. To simplify things, every test got their own capital letter. Test A, B, C, D and E. Information of preparations, material content and days of hardening of each

sample is described in Table C1 in Appendix C.

Test A - Mixing efficiency

The most efficient way to utilize the pozzolanic properties in a concrete, is to mix the pozzolana and the cement as thoroughly as possible. This is best achieved by milling the components in a ball mill for a short period [2].

To check if the milling had any effect on the compressive strength, two mixtures (AP/IC and FA/IC) were milled lightly together one at a time in a Los Angeles Mill. We decided that 15 test cubes of each mixture were necessary to execute wanted tests. Used amount of materials and water is described in Table B2 and B3 in Appendix B. As a reference an un-milled AP/IC mixture was used.

Test B – Accelerated hardening

A rule of thumb says that for every 10 °C increase the hardening doubles. By increasing the water temperature from 20°C to 40°C, should thus increase the strength process four times.

In this test we used six of the cement cubes that were casted in Test A. Three cubes of each mixture. After one day at room temperature the cubes were placed in a 40 °C curing bath for six days. Temperatures higher than 40 °C could harm the hardening process. Because of water evaporation, more water had to be added regularly. The cubes were compression tested after total 7 hardening days. As a reference the cubes after total 28 hardening days at 20°C were used.

If the accelerated hardening corresponds to a rule of thumb for the hardening day/strength ratio, it could be used later in this thesis for longer hardening possesses.

Test C – Milled components together and particle size reduced (<20 µm)

Unless the pozzolana is already in a fine powdered form, it should be ground in a ball or rod mill. The greater amount of milling, the finer the pozzolana will become and rate of reaction will increase. The pozzolana and cement must be mixed as thoroughly as possible [2].

Test C was done to see if reduced grain size of aplite and cement had any influence on compressive strength. Aplite and industry cement were milled in a Retsch Planetary Ball Mill PM 100 for approximately 30 minutes. After that the mixture was placed in a sieving machine. The machine was used to scalp the mixture to particles smaller than 20 µm. Remaining particles larger than 20 µm were milled one more time. Sieving and milled was utilized simultaneously until wanted amount was achieved. Six cubes were casted. Used mixture ratio is listed in Table B4 in Appendix B. As a reference milled AP/IC cubes were used.

Test D – Reduced aplite grain size (40µm)

To function properly, the pozzolana must be finer than 45 µm (microns) in particle size. Finer particle sizes are more quickly converted to a supplementary cementitious material [13].

This test had the same intensions as Test C, to see if the grain size reduction had any influence on the compressive strength. The difference was that only the aplite was milled. According to the measured aplite particle distribution, 58.11% of the sample passed the 40µm sieve. Measured particle size distribution for the aplite is given in Table A1 in Appendix A.

The aplite was milled in the Retsch Planetary Ball Mill PM 100 for approximately 30 minutes. Then it was placed in the sieving machine. Only the 40µm and 125µm sieve was used. The sample was divided into two parts that contained particles larger and smaller than

40µm. This was done until necessary amount was achieved. Industry cement and water was added. Six cubes of each mixture were casted. The mixture composition is described in Table B4 in Appendix B.

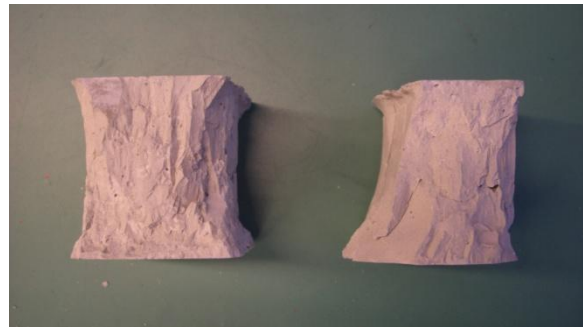
Test E – Reduced aplite grain size (<20µm)

Test E had the same intensions and procedures as Test D. The 40µm sieve was exchanged to a 20µm sieve. Sieved particles larger than 20 µm were milled one more time. This process was utilized until necessary amount was achieved. Industry cement and water was mixed in before casting. Six cubes were casted. The mixture composition was the same as in Test D. As a reference the same reference cubes as in Test D were used.

A set of test cubes containing only neat industry cement were casted. Used amount of industry cement and water is given in Table B5 in Appendix B.

Sample testing

To test the cement cubes compressive strength, a Toni Tech machine was utilized. A guiding booklet was used to assure that the testing was carried out correctly and secure. A metallic cube with the same size as the test cubes was placed in the centre of the machine. One sample was pressed at a time. The test cubes were picked up from the curing bath and wiped clean before being placed on the top of the metallic cube. Each cube was pressed to fracture. The machines loading speed gave a pressure increase of 0.5 N/mm²s. Each sample was manually checked to verify the fracture type and form. Samples with wrong fracture form were noted and declared inapplicable. The compression testing was executed according to NS EN 12390-3 [14].



*Figure 6: Cement cubes fracture forms.
(left-hand side sample 11-1, right-hand side sample 11-2)*

Fig. 6 shows cement cubes that are pressed to fracture. Test cube 11-1 at the left hand side indicates a valid fracture form. Little or nothing of the surfaces contacted with the press machine is damaged. Test cube 11-2 at the right hand side shows an invalid fracture form. One third of the upper pressed surface is off. Cubes with invalid fracture form are ignored in mean value calculations.

RESULTS AND DISSCUSION

The information and the compressive strength of each cement cube have been gathered in Appendix C.

The bar charts below are mean values of each test series. A cube which stands out with considerable lower compressive strength in proportion to the others in a test series is ignored in mean value calculations. If the test cubes compressive strength varies 20% from the other test cubes mean value in a series, the cube is invalid.

If all the cubes compressive strengths in a test series vary just as much, all tests are used in mean value calculations.

The different test series have an ID defined with a number, 1 to 22. Every cube is marked with a number behind the test series number to simplify the identification. The compressive strength is given in MPa.

The 20°C curing bath had no thermostat. The temperature in the curing

bath was measured regularly. Lowest water temperature was measured to 14.6°C. This may have had some effect on the test results.

Test A - Mixing efficiency

Fig. 7 shows a bar chart with compressive strength for milled AP/IC and FA/IC samples. Here the mean values from test series 1, 2, 5, 6, 7, 8, 9 and 10 are shown. The AP/IC cubes gained 22.08, 39.52, 53.19 and 56.28 MPa after 1, 7, 28 and 77 days respectively. The FA/IC cubes show generally higher strength than the AP/IC cubes. The FA/IC gained 7.13, 7.5, 6.52 and 19 MPa more than the AP/IC after respectively 1,7,28 and 77 days.

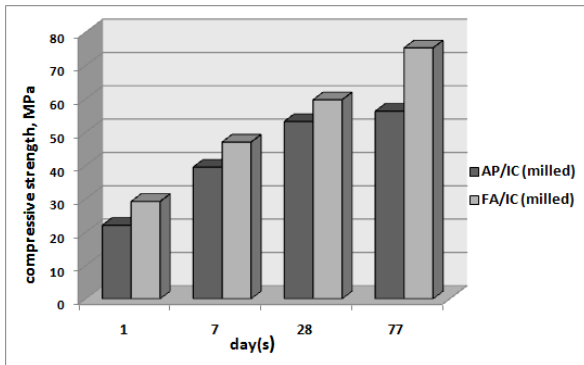


Figure 7: Compressive strength for milled AP/IC and FA/IC cubes.

The strength ratio between the mixtures seems to be stable, except the 77 days testing. The FA/IC cubes tested after 77 days shows a significantly higher compressive strength than the AP/IC cubes. In the early testing some FA/IC cubes failed and lead to too few cubes for the last test. Only one FA/IC cube was tested after 77 days, which means that the data is statistically poor.

To see if the milling of aplite and industry cement had any influence on the compressive strength, milled and un-milled AP/IC cubes were compared. This comparison is illustrated in Fig. 8. Mean values from test series 6, 8, 17 and 18 have

been used. The un-milled AP/IC cubes show a compressive strength mean value on 42.37 and 54.10 MPa after respectively 7 and 28 days. For the bar charts in the rest of this thesis, un-milled AP/IC is shown as just AP/IC.

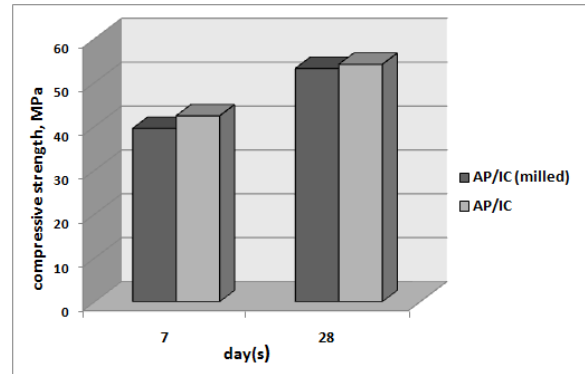


Figure 8: Compressive strength for milled and un-milled AP/IC cubes.

An important note is that two different industry cement packages have been used. The un-milled AP/IC contained the newest industry cement. Yet the milling did not seem to have any worth mentioning influence on the compressive strength. The milled AP/IC had a lower compressive strength compared to the one that was found for un-milled. This may be due to the cement was old, had reacted some with CO₂ and was also pre-hydrated to some extent.

Test B – Accelerated hardening

Fig. 9 shows compressive strength for FA/IC and AP/IC cubes placed in two different curing baths. Here the mean values from test series 3, 4, 7 and 8 have been used. The mean values from the test cubes at the 40°C curing bath and tested after total 7 hardening days shows 40.92 and 54.59 MPa for respectively AP/IC and FA/IC. After total 28 hardening days, the cubes in the 20°C curing bath gained 53.32 and 59.71 MPa compressive strength.

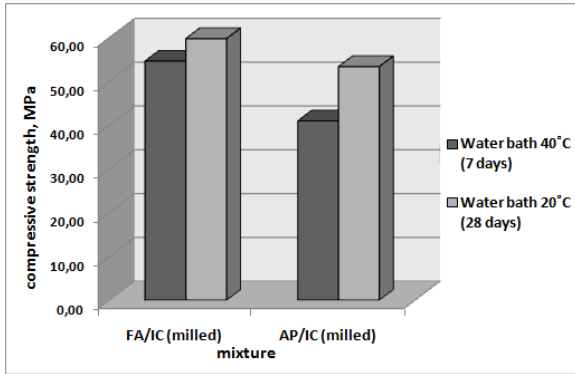


Figure 9: Compressive strength for FA/IC and AP/IC cubes in different curing baths.

The test cubes which were placed in a 20°C curing bath show a higher compressive strength than the cubes which were placed in the 40°C curing bath. As much as 30.3% strength difference for the AP/IC cubes. The hypothesis fail and was not used further in this thesis.

To see if the increased temperature had any effect at all, cement cubes that was tested after total 7 hardening days were compared. Fig. 10 shows the compressive strength for FA/IC and AP/IC cubes after total 7 hardening days in different curing baths. The mean values from test series 3, 4, 5 and 6 have been used.

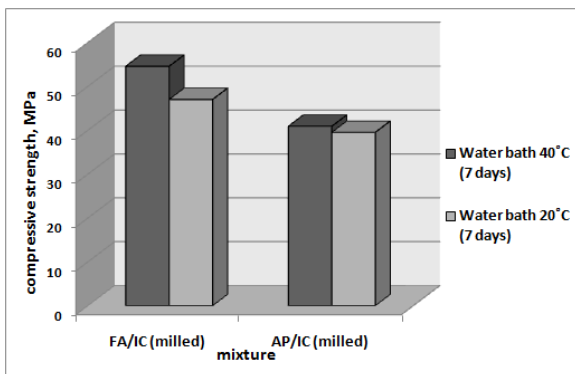


Figure 10: Compressive strength for FA/IC and AP/IC cubes after total 7 hardening days in different curing baths.

As the bar graph indicates, the curing bath had no specific influence on the AP/IC cubes compressive strength. The FA/IC

cubes on the other hand, gained 16.1% more strength in the 40°C curing bath.

Test C – Milled components together and particle size reduced (<20 μm)

Test C was done by combining particles size decreasing and milling aplite and industry cement together. Fig. 11 shows the compressive strength for AP/IC cubes which contained component particles below 20 μm. The mean values from test series 15, 16 and 23 are shown. The cubes gained 41.38, 51.18 and 61.72 MPa compressive strength after 7, 28 and 72 days respectively.

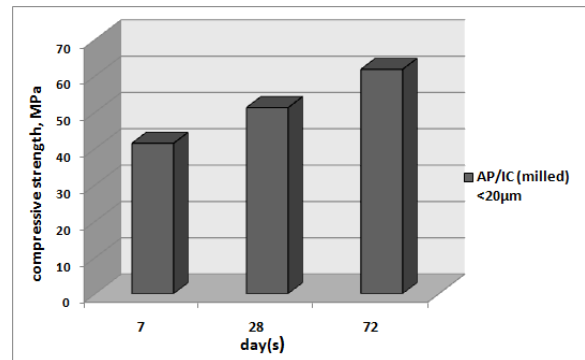


Figure 11: Compressive strength for AP/IC cubes with component particles below 20 μm.

Lack of time made it difficult to make more cement mixture than for six test cubes. Only one and two cubes were tested after 72 and 28 days respectively.

To see if the decreased component particles to below 20 μm had any effect at all, milled AP/IC cubes were used as comparison. Fig. 12 shows compressive strength for AP/IC cubes which are milled below 20 μm and AP/IC cubes which are lightly milled together. The mean values from test series 6, 8, 15 and 16 are shown.

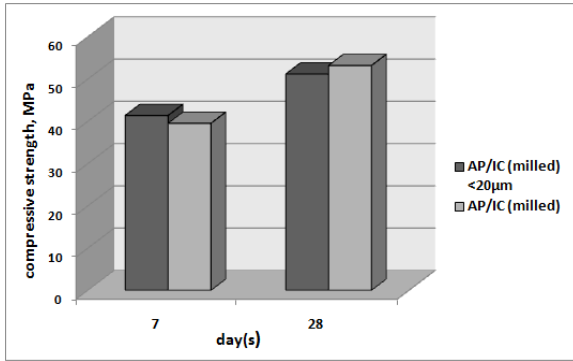


Figure 12: Compressive strength for AP/IC cubes that contains components milled below 20 µm and cubes that contains a lightly milled mixture.

The mixture with component particles below 20 µm got dry and uneven after been mixed with water. More water had to be mixed in. This increased the w/c ratio from 0.38 to 0.43. Increased w/c ratio leads to decreased compressive strength. The cement mixture seemed to need more water as the grain size decreased. Increased fineness gives a faster heat generation and solidity development caused by the increased contact surface between cement and water. Industry cement has already been milled to reduce the grain size and setting time. With different w/c ratio, the compression strength comparison is not good. Despite the increased w/c ratio, it was not found that particle size decreasing gains extra strength.

Test D – Reduced aplite grain size (40 µm)

In Test D was milled aplite divided into two parts. The cement and the aplite were not milled together before making the concrete mixture. When adding water the slurry behaved well. The slurry was not dry and uneven as in Test C. This was avoided by not reducing the cements particles.

Fig. 13 shows compressive strength for AP/IC cubes with different aplite particle sizes. Mean values from test series 11, 12, 13 and 14 are shown. The AP/IC cubes that contained aplite below 40 µm gained 49.51 and 61.63 MPa after 7 and 28 days respectively. The bar chart also

showed that the cubes which contained aplite above 40 µm gained 52.83 and 56.11 MPa after 7 and 28 days respectively.

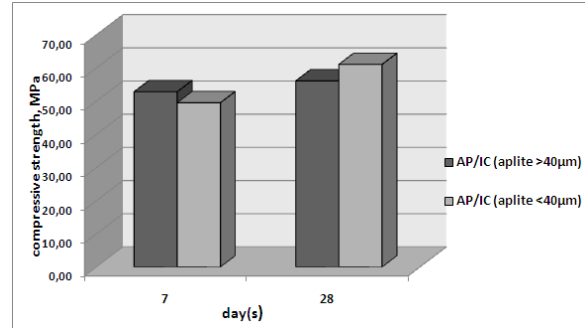


Figure 13: Compressive strength for AP/IC cubes with different aplite particle sizes.

The difference in compressive strength between the mixtures after 7 days is 6.7%. After 28 days on the other hand, the cubes with aplite below 40 µm gained 8.8% more strength than the cubes with aplite particles above 40 µm. This may indicate that particle size reduction has a positive effect between 7 and 28 days.

When calculating the mean values, some of the cubes were excluded because of too low compressive strength. Cube 12-1 gained 19.3% less strength than the mean value from cube 12-2 and 12-3.

On the other hand, the particle size distributions of the milled aplite samples are not known. The aplite mixture with particles above 40 µm could contain particles below 20 µm, since the sieving/scalping was only done for a half hour.

Test E – Reduced aplite grain size (20 µm)

In this test the aplite was milled down to particles below 20 µm. Fig. 14 shows compressive strength for AP/IC cubes with aplite particles below 20 µm. The mean values from test series 17, 18, 20 and 22 are shown. The AP/IC cubes with aplite below 20 µm gained 54.30 and 70.06 MPa after 7 and 28 days respectively.

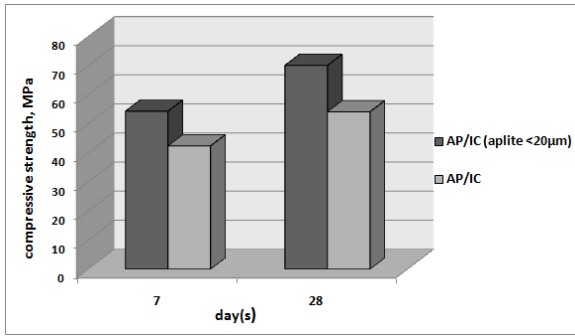


Figure 14: Compressive strength for AP/IC cubes with aplite particles below 20 μm and un-milled AP/IC cubes.

The AP/IC cubes with aplite particles below 20 μm gained 28.1 and 29.5 % more after 7 and 28 days respectively than the un-milled AP/IC cubes. The difference in percent seems to be stable. This may indicate that the decreased particles react more rapid than the larger ones.

Concrete cubes with neat industry cement and 0.38 w/c ratio were made for comparison. Fig. 15 shows AP/IC cubes with aplite particles below 20 μm and neat industry cement cubes. Here the mean values from test series 19, 20, 21 and 22 are shown. The neat cement test cubes gained 56.11 and 63.77 MPa after 7 and 28 days respectively.

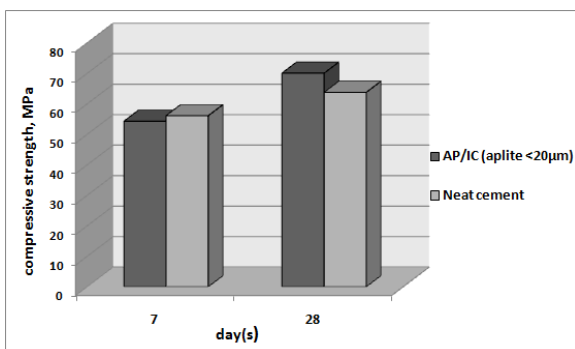


Figure 15: Compressive strength for AP/IC cubes with aplite particles below 20 μm and neat industry cement cubes.

The neat industry cement gained only 3.3 % more strength after 7 days

hardening. After 28 days the AP/IC cubes gained 9.9% more strength than the neat industry cement. From 7 to 28 days the AP/IC cubes increased the compressive strength with 29%.

The neat cements compressive strength is a bit lower than standardized values using a 0.40 w/c ratio. This may be due to, a too low w/c ratio, inhomogeneous mixing or low curing bath temperature.

Fig. 15 is made to get a better overview of the particle size reduction for aplite. It shows AP/IC cubes with varying particle sizes, with ascending order from left to right.

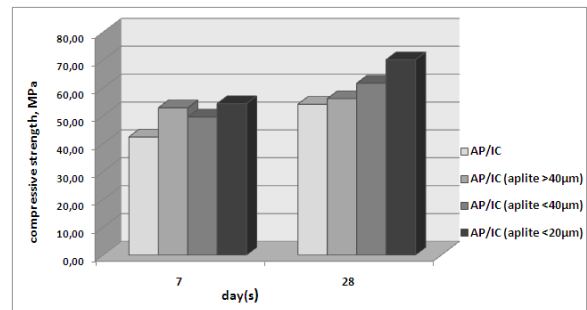


Figure 15: Compressive strength for AP/IC cubes with difference particle size distribution.

The tendency clearly indicates increased compressive strength; the finer the particles are milled.

To find the particle size distribution for AP/IC cubes with aplite particles below 20 μm, the AcoustoSizer II was utilized. The test showed a particle size distribution having a d15 of 0.038 μm, a d50 of 0.066 μm and a d85 of 0.183 μm. This was much finer particles than expected. Graphic charts are shown in Fig. A5-A8 in Appendix A.

CONCLUSIONS

Following conclusions may be drawn from the present study:

Milling aplite and industry cement together gained no strength. The fly ash and industry cement may have had some effect of the milling.

The increased temperature in the water curing had little effect AP/IC test cubes.

Milling the cement and the aplite together and thus reducing the particle size to below 20 μm seemed to have little effect on the compressive strength.

The results indicate increased compressive strength on AP/IC cubes with decreased aplite particles. Especially the test cubes with aplite below 20 μm , shows a great improvement in strength from 7 to 28 days.

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- [11] <http://www.retsch.com/products/milling/planetary-ball-mills/pm100/>
- [12] NS-EN 12390-2 Testing hardened concrete Part 2: Making and curing Specimens for strength tests.
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APPENDIX A – PARTICLE SIZE DISTRUBUTION CHARTS



Syrpatee GmbH
System-Partikel-Technik

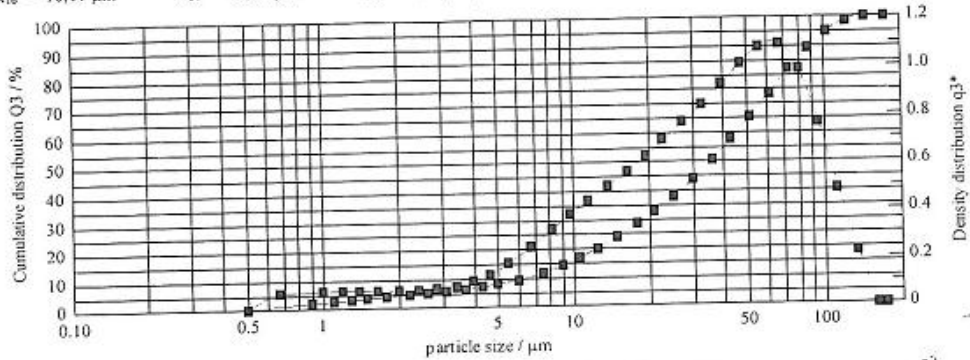
HELOS Particle Size Analysis
WINDOX 5

HELOS (H1223) & QUIXEL, R3: 0.5/0.9...175µm 2008-10-17, 15:28:27,7021

Quartz

WARNING: Coarse particles probably exceeding the measuring range.

$x_{10} = 6,58 \mu\text{m}$ $x_{50} = 35,74 \mu\text{m}$ $x_{90} = 89,52 \mu\text{m}$ SMD = 11,19 µm VMD = 42,66 µm
 $x_{16} = 10,17 \mu\text{m}$ $x_{84} = 77,38 \mu\text{m}$ $x_{99} = 132,55 \mu\text{m}$ $S_v = 0,54 \text{ m}^3/\text{cm}^3$ $S_m = 2024,12 \text{ cm}^3/\text{g}$



comment:

user parameters:

P1: FOvH
P2: Heli Utvikling AS
P3: 10 - 100 mikron produkt

cumulative distribution

| $x_0/\mu\text{m}$ | $Q_3/\%$ | $x_0/\mu\text{m}$ | $Q_3/\%$ | $x_0/\mu\text{m}$ | $Q_3/\%$ | $x_0/\mu\text{m}$ | $Q_3/\%$ |
|-------------------|----------|-------------------|----------|-------------------|----------|-------------------|----------|
| 0,90 | 1,58 | 3,70 | 6,09 | 15,00 | 23,83 | 61,00 | 73,21 |
| 1,10 | 2,19 | 4,30 | 6,78 | 18,00 | 28,28 | 73,00 | 81,66 |
| 1,30 | 2,71 | 5,00 | 7,66 | 21,00 | 32,45 | 87,00 | 89,13 |
| 1,50 | 3,16 | 6,00 | 9,08 | 25,00 | 37,64 | 103,00 | 94,66 |
| 1,80 | 3,72 | 7,50 | 11,46 | 30,00 | 43,66 | 123,00 | 98,34 |
| 2,20 | 4,34 | 9,00 | 14,01 | 36,00 | 50,28 | 147,00 | 100,00 |
| 2,60 | 4,86 | 10,50 | 16,56 | 43,00 | 57,39 | 175,00 | 100,00 |
| 3,10 | 5,43 | 12,50 | 19,88 | 51,00 | 64,84 | | |

density distribution (log.)

| $x_m/\mu\text{m}$ | q_3/g | $x_m/\mu\text{m}$ | q_3/g | $x_m/\mu\text{m}$ | q_3/g | $x_m/\mu\text{m}$ | q_3/g |
|-------------------|----------------|-------------------|----------------|-------------------|----------------|-------------------|----------------|
| 0,67 | 0,06 | 3,39 | 0,09 | 13,69 | 0,50 | 55,78 | 1,08 |
| 0,99 | 0,07 | 3,99 | 0,11 | 16,43 | 0,56 | 66,73 | 1,08 |
| 1,20 | 0,07 | 4,64 | 0,13 | 19,44 | 0,62 | 79,69 | 0,98 |
| 1,40 | 0,07 | 5,48 | 0,18 | 22,91 | 0,69 | 94,66 | 0,75 |
| 1,64 | 0,07 | 6,71 | 0,25 | 27,39 | 0,76 | 112,56 | 0,48 |
| 1,99 | 0,07 | 8,22 | 0,32 | 32,86 | 0,84 | 134,47 | 0,21 |
| 2,39 | 0,07 | 9,72 | 0,38 | 39,34 | 0,92 | 160,39 | 0,00 |
| 2,84 | 0,08 | 11,46 | 0,44 | 46,83 | 1,01 | | |

evaluation: WINDOX 5.2.0.0, HRLD

revalidation:
reference measurement: 10-17 15:27:24
contamination: 0,00 %

product: Quartz

density: 2,65 g/cm³
shape factor: 1,00
 $C_{opt} = 22,78 \%$

trigger condition: 10s (QUIXEL, SUCCELL, CUVETTE)b

time base: 1000,00 ms
start: button
valid: always
stop: 10s real time

dispersing method: QUIXEL SIC-P80, 6mm
dispersant: Water+NA4P2O7, temperature: 25,00 °C
size of cuvette: 6 mm
sonication duration: 0,00 s, pause: 0,00 s
pump speed: 40,00 %

2008-10-17 15:28:38

User

Page 1

Figure A1: Specification sheet for aplite

Measured particle sizes

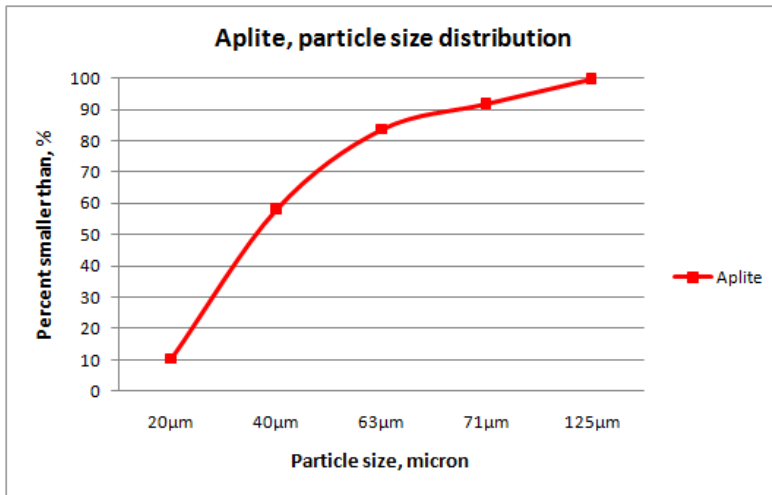


Figure A2: Measured particle size distribution for aplite.

Table A1: Aplite, percent passing.

| Mesh size | Percent passing |
|-------------|-----------------|
| 20 μ m | 10.42% |
| 40 μ m | 58.11% |
| 63 μ m | 83.64% |
| 71 μ m | 91.93% |
| 125 μ m | 99.78% |
| Total | 100% |

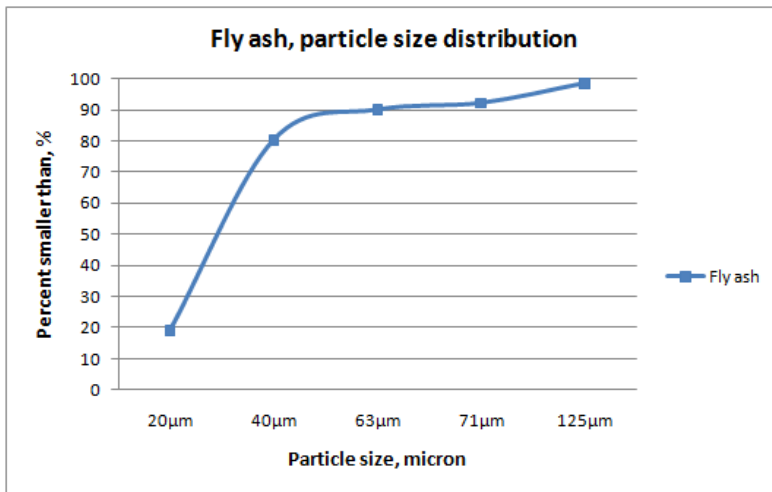


Figure A3: Measured particle size distribution for fly ash.

Table A2: Fly ash, percent passing.

| Mesh size | Percent passing |
|-------------|-----------------|
| 20 μ m | 19.13% |
| 40 μ m | 80.34% |
| 63 μ m | 89.96% |
| 71 μ m | 92.09% |
| 125 μ m | 98.46% |
| Total | 100% |

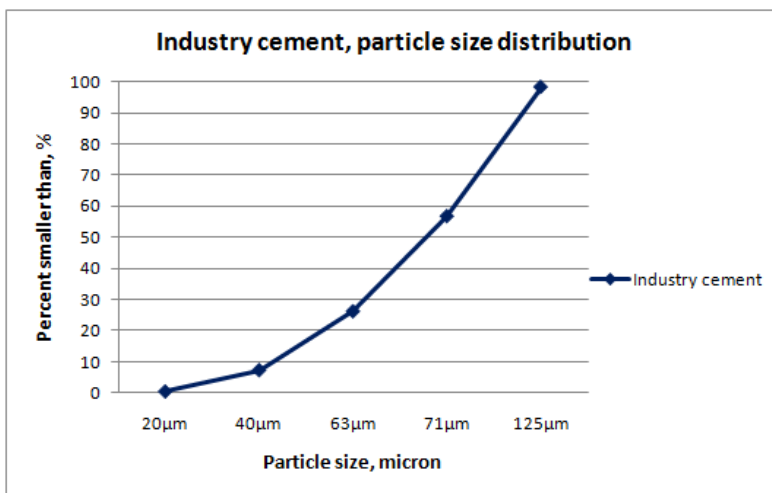


Figure A4: Measured particle size distribution for industry cement

Table A3: Industry cement, percent passing.

| Mesh size | Percent passing |
|-------------|-----------------|
| 20 μ m | 1.66% |
| 40 μ m | 43.18% |
| 63 μ m | 73.78% |
| 71 μ m | 92.68% |
| 125 μ m | 99.56% |
| Total | 100% |

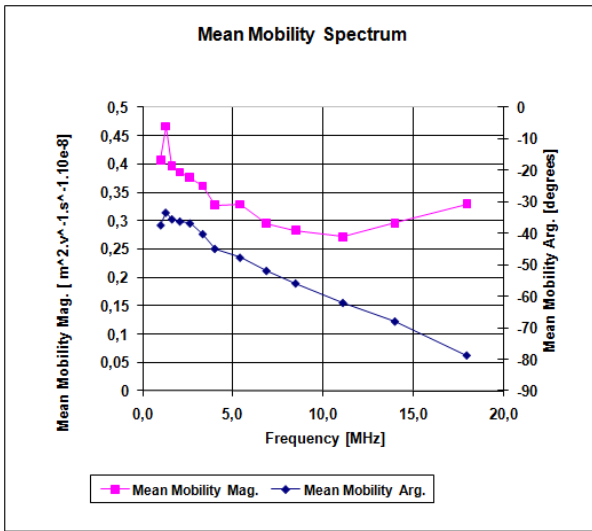


Figure A5: Mean mobility spectrum

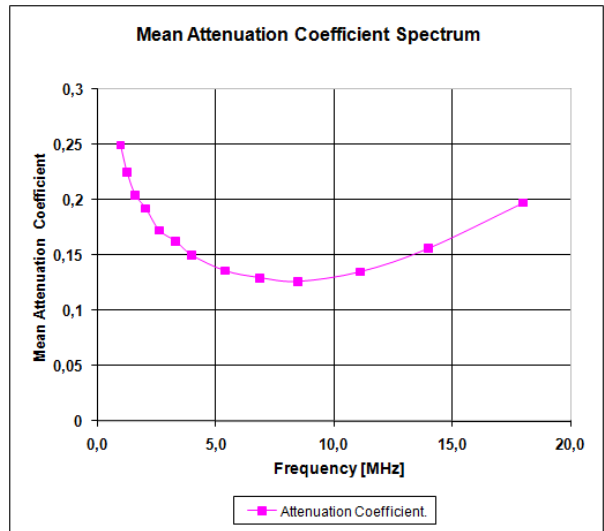


Figure A6: Mean attenuation coefficient spectrum

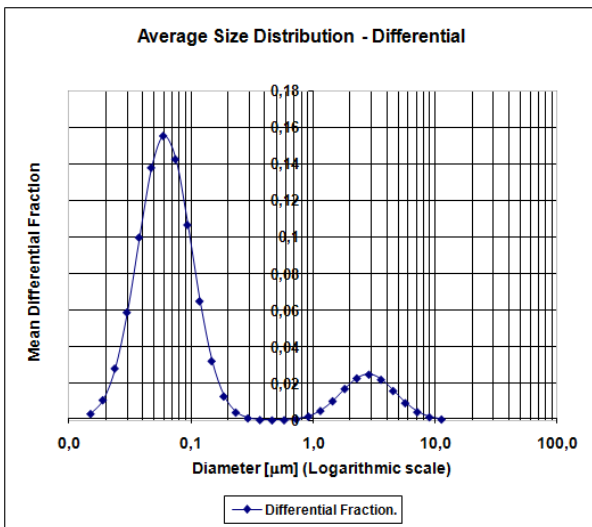


Figure A7: Average size distribution - differential

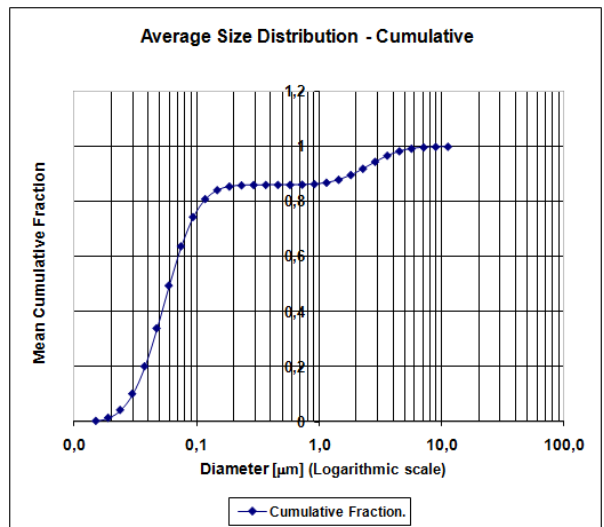


Figure A8: Average size distribution - cumulative

APPENDIX B – TEST CUBE PROPORTION

Density measurement

Table B1: Density measurements for aplite

| | Formula | Test 1 | Test 2 |
|------------------|---|-----------------------|-----------------------|
| Volumetric flask | - | 231.78 g | 231.75 g |
| Sample | - | 139.91 g | 59.56 g |
| Water | - | 943.98 g | 973.43 g |
| Water2 | $1000 \text{ cm}^3 - \text{Water cm}^3$ | 56.02 cm^3 | 26.57 g |
| Density | $\text{Sample g} / \text{Water2 cm}^3$ | 2.50 g/cm^3 | 2.24 g/cm^3 |

Cement proportion

Table B2: Fly ash/ industry cement (FA/IC) mixture for 15 test cubes.

| Type | Weight | Weight % | Volume % | Volume |
|--|----------|----------|----------|----------------------|
| FA | 0.573 kg | 13.275 % | 20 % | 0.300 dm^3 |
| IS | 3.744kg | 86.725 % | 80 % | 1.200 dm^3 |
| Total | 4.317 kg | 100 % | 100 % | 1.500 dm^3 |
| Amount of water: $0.38 \times 4.317 \text{ kg} = 1.640 \text{ kg}$ | | | | |

Table B3: Aplite/ industry cement (AP/IC) mixture for 15 test cubes.

| Type | Weight | Weight % | Volume % | Volume |
|--|----------|----------|----------|----------------------|
| AP | 0.795 kg | 17.51 % | 20 % | 0.300 dm^3 |
| IS | 3.744 kg | 82.49 % | 80 % | 1.200 dm^3 |
| Total | 4.539 kg | 100 % | 100 % | 1.500 dm^3 |
| Amount of water: $0.38 \times 4.539 \text{ kg} = 1.725 \text{ kg}$ | | | | |

Table B4: Aplite/ industry cement (AP/IC) mixture for 6 test cubes.

| Type | Weight |
|-------|----------|
| AP | 397.5 g |
| IS | 1872.0 g |
| Water | 862.4 g |

Table B5: Neat industry cement (IC) mixture for 6 test cubes.

| Type | Weight |
|-------|--------|
| IC | 2000 g |
| Water | 760 g |

APPENDIX C – TENSILE STRENGTH RESULTS

Table C1: Tensile strength for all test cubes.

* Samples were placed in a 40 °C curing bath for 6 days and compression tested after 7 days.

** Sample 7-2 had a defect and broke down early in the compression test. The cube was therefore excluded from the mean value calculation.

*** The samples were excluded cause of too low values versus the other tests.

**** Samples with invalid fracture form.

| ID | Pozzolana type | Milled | Grain size | Tested [days] | Strength [MPa] | Mean value [MPa] | Cement |
|----------|----------------|--------|------------|---------------|----------------|------------------|---------|
| 1-1 | fly ash | yes | normal | 1 | 30.16 | 29.21 | 1. type |
| 1-2 | fly ash | yes | normal | 1 | 30.48 | | 1. type |
| 1-3 | fly ash | yes | normal | 1 | 27.98 | | 1. type |
| 1-4 | fly ash | yes | normal | 1 | 28.20 | | 1. type |
| 2-1 | aplite | yes | normal | 1 | 22.12 | 22.08 | 1. type |
| 2-2 | aplite | yes | normal | 1 | 22.05 | | 1. type |
| 2-3 | aplite | yes | normal | 1 | 22.06 | | 1. type |
| 3-1 | fly ash | yes | normal | 7* | 58.22 | 54.59 | 1. type |
| 3-2 | fly ash | yes | normal | 7* | 47.90 | | 1. type |
| 3-3 | fly ash | yes | normal | 7* | 57.64 | | 1. type |
| 4-1 | aplite | yes | normal | 7* | 40.15 | 40.92 | 1. type |
| 4-2 | aplite | yes | normal | 7* | 40.49 | | 1. type |
| 4-3 | aplite | yes | normal | 7* | 42.13 | | 1. type |
| 5-1 | fly ash | yes | normal | 7 | 45.42 | 47.02 | 1. type |
| 5-2 | fly ash | yes | normal | 7 | 45.49 | | 1. type |
| 5-3 | fly ash | yes | normal | 7 | 50.16 | | 1. type |
| 6-1 | aplite | yes | normal | 7 | 39.95 | 39.52 | 1. type |
| 6-2 | aplite | yes | normal | 7 | 39.98 | | 1. type |
| 6-3 | aplite | yes | normal | 7 | 38.64 | | 1. type |
| 7-1 | fly ash | yes | normal | 28 | 60.02 | 59.71 | 1. type |
| 7-2** | fly ash | yes | normal | 28 | 25.51 | | 1. type |
| 7-3 | fly ash | yes | normal | 28 | 64.51 | | 1. type |
| 7-4 | fly ash | yes | normal | 28 | 54.61 | | 1. type |
| 8-1 | aplite | yes | normal | 28 | 52.02 | 53.19 | 1. type |
| 8-2 | aplite | yes | normal | 28 | 54.23 | | 1. type |
| 8-3 | aplite | yes | normal | 28 | 53.32 | | 1. type |
| 9-1 | fly ash | yes | normal | 77 | 75.28 | 75.28 | 1. type |
| 10-1*** | aplite | yes | normal | 77 | 43.24 | 56.28 | 1. type |
| 10-2 | aplite | yes | normal | 77 | 58.94 | | 1. type |
| 10-3 | aplite | yes | normal | 77 | 53.61 | | 1. type |
| 11-1 | aplite | no | >40 µm | 7 | 53.65 | 52.83 | 2. type |
| 11-2**** | aplite | no | >40 µm | 7 | 44.54 | | 2. type |

| | | | | | | | |
|---------|-------------|-----|-------------------|----|-------|-------|---------|
| 11-3 | aplite | no | >40 μm | 7 | 52.00 | | 2. type |
| 12-1 | aplite | no | >40 μm | 28 | 48.39 | 56.11 | 2. type |
| 12-2 | aplite | no | >40 μm | 28 | 60.06 | | 2. type |
| 12-3 | aplite | no | >40 μm | 28 | 59.87 | | 2. type |
| 13-1 | aplite | no | <40 μm | 7 | 51.22 | 49.51 | 2. type |
| 13-2 | aplite | no | <40 μm | 7 | 48.52 | | 2. type |
| 13-3 | aplite | no | <40 μm | 7 | 48.78 | | 2. type |
| 14-1 | aplite | no | <40 μm | 28 | 60.39 | 61.63 | 2. type |
| 14-2 | aplite | no | <40 μm | 28 | 63.44 | | 2. type |
| 14-3 | aplite | no | <40 μm | 28 | 61.07 | | 2. type |
| 15-1 | aplite | yes | <20 μm | 7 | 47.47 | 41.38 | 1. type |
| 15-2 | aplite | yes | <20 μm | 7 | 40.48 | | 1. type |
| 15-3 | aplite | yes | <20 μm | 7 | 36.19 | | 1. type |
| 16-1 | aplite | yes | <20 μm | 28 | 49.67 | 51.18 | 1. type |
| 16-2 | aplite | yes | <20 μm | 28 | 52.69 | | 1. type |
| 17-1 | aplite | no | normal | 7 | 37.60 | 42.37 | 2. type |
| 17-2 | aplite | no | normal | 7 | 45,60 | | 2. type |
| 17-3 | aplite | no | normal | 7 | 43,90 | | 2. type |
| 18-1 | aplite | no | normal | 28 | 52.75 | 54.10 | 2. type |
| 18-2 | aplite | no | normal | 28 | 55.00 | | 2. type |
| 18-3 | aplite | no | normal | 28 | 54.56 | | 2. type |
| 19-1 | neat cement | no | normal | 7 | 60.92 | 56.11 | 2. type |
| 19-2 | neat cement | no | normal | 7 | 52.8 | | 2. type |
| 19-3 | neat cement | no | normal | 7 | 54.6 | | 2. type |
| 20-1 | aplite | no | <20 μm | 7 | 49.33 | 54.30 | 2. type |
| 20-2 | aplite | no | <20 μm | 7 | 54.33 | | 2. type |
| 20-3 | aplite | no | <20 μm | 7 | 59.23 | | 2. type |
| 21-1 | neat cement | no | normal | 28 | 61.49 | 63.77 | 2. type |
| 21-2*** | neat cement | no | normal | 28 | 48.00 | | 2. type |
| 21-3 | neat cement | no | normal | 28 | 66.05 | | 2. type |
| 22-1 | aplite | no | <20 μm | 28 | 70.17 | 70.06 | 2. type |
| 22-2 | aplite | no | <20 μm | 28 | 68.10 | | 2. type |
| 22-3 | aplite | no | <20 μm | 28 | 71.37 | | 2. type |
| 23-1 | aplite | yes | <20 μm | 72 | 61.73 | 61.73 | 1. type |