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Work package 1 - Laboratory testing results

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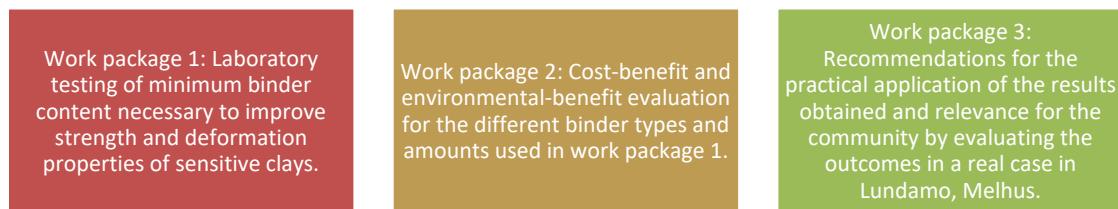
Review and reference page

1 Introduction

Research at NGI has shown that it is possible to reduce the binder content necessary to improve soil strength and deformation properties of sensitive clays. However, recent results indicate that there is a lower limit to the binder content required to get this enhancing process started. Further laboratory testing is needed to define this lower limit (i.e. minimum amount) of binder and to quantify the environmental impact of this reduction in terms of CO₂-emissions. Additionally, the practical evaluation of binder reduction needs to be assessed in a real application case.

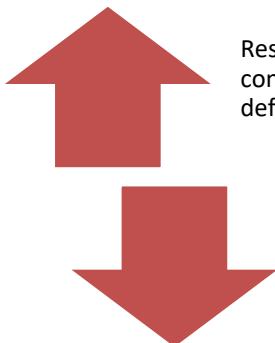
The project SUstainable Soil Improvement (SUSI) aims to find the most sustainable and economical amount and type of binder to improve strength and deformation properties of sensitive clays. SUSI is a research collaboration project between Melhus municipality (project owner), NGI (project leader), Franzefoss Minerals and JLE Grunnforsterkning; financed by RFF Trøndelag through the grant 310057.

The project is divided in three work packages:



The present technical note summarizes the work done in the work package 1.

2 Description of WP1



Research question: What is the minimum binder content necessary to improve strength and deformation properties of sensitive clays?

Proposed solution: Collection of clay samples from NGTS Tiller-Flotten and laboratory tests with different lime-cement (binder) types and amounts, to find the minimum amount necessary to improve strength and deformation properties of sensitive clays.

The clay for laboratory tests was taken from the quick clay Norwegian GeoTest Site (NGTS) at Tiller-Flotten, Trondheim. Tiller-Flotten is one of the five geotest sites established in 2016 with support from The Research Council of Norway (RCN) and collaborating partners with the purpose of being used as field laboratories for development, testing and verification of new innovative methods for site investigations

and testing procedures. The Tiller-Flotten quick clay has been used before for testing different binders in other projects.

SUSI project deals with testing of specific lime products which are locally available in Trøndelag. Other binder products, like cement kiln dust (CKD) or alternatives like ashes are not included in the project. The different types of burnt lime combined with the same cement type were tested to see if there is a difference between the binder mixtures used. A mixture of lime-cement in a 50/50 ratio was used. The binder content was varied around limit values observed in previous results obtained and summarized in NGI (2019). The strength and deformation properties of the stabilized clay were measured in unconfined compression tests after 28 days of curing. The samples were cured at room temperature.

3 Experimental work

3.1 Burnt lime products

Experiments have been made with four different burnt lime-based binders: LKD, Stabila B60, Stabila B80 and Stabila B100. What distinguishes the various Stabila products is the content of LKD (Lime Kiln Dust), which is a residual product formed by the combustion of limestone. Stabila B100 contains pure burnt lime, which consists mainly of calcium oxide (CaO). Stabila B80 contains 15-20% LKD, while Stabila B60 contains 20-40% LKD. The remaining percentage consists of burnt lime. A flow agent is also added during the production of Stabila B60, B80 and B100.

Stabila B100 satisfies the requirements for CL 90-Q in NS-EN 459. B60 and Stabila B80 satisfy the requirements for CL 70-Q and CL 80-Q in NS-EN 459, respectively. The chemical composition of LKD varies with its production process and depends on, for example, the type of limestone is burned and what fuel is used (Latif et al., 2015). LKD from Franzefoss Minerals contains mainly burnt lime and calcium carbonate (CaCO_3).

Table 1 shows the chemical composition and reactivity (t_{60}) in the various Stabila products. See Appendix A for a complete analysis report.

Table 1 Overview of constituents in the different types of lime

Product	CaO + MgO [%]	CaO-active [%]	Reactivity NS-EN 495	
			t_{60} [sec]	ΔdT [$^{\circ}\text{C}$]
LKD	>70	<30	n.a.	<20
Stabila B60 (CL 70-Q)	>90	75 ± 5	>500	>40
Stabila B80 (CL 80-Q)	<96	85 ± 5	>180	<50
Stabila B100 (CL 90-Q)	>96	95 ± 5	<180	>50

3.2 Cement

The cement used is a standard cement FA (CEM II/B-M) according to NS-EN 197-1, Kjøpsvik. It is composed by 78% clinker, 18% fly ash, 4% limestone, less than 4% sulfate (SO_3), 1,5% alkalies (Na_2O) and less than 0,085% chloride. These values according to the product data sheet from Norcem (last revised in August 2017).

3.3 Clay

The clay is taken from the Norwegian GeoTest Site (NGTS) for quick clay at Tiller-Flotten, Trondheim. The site is relative homogeneous and well described by L'Heureux et al. (2019). The samples were block samples from the borehole TILB19 from depth ranging between 8,5-8,9 m and 8,9-9,3 m. The clay is a marine clay with a water content of 45%, unit weight near 18 kN/m³, plasticity index of 15% ($\text{WP} = 21\%$ og $\text{WL} = 36\%$), salt content (NaCl) of 2 g/l and a clay content near 68% (and a silt content of 32%), all these values around the sampling depth.

Data provided by NGTS project describes that the clay mineralogy of Tiller-Flotten quick clay is dominated in the clay fraction (< 2 μm) by biotite (26%) and illite / muscovite (23%), followed by chlorite (16%), plagioclase (13%), amphibolite (7%), quartz (7%) and potassium feldspar (4-6%). In the bulk fraction, the clay has 21% illite, 19% quartz, 19% biotite, 17% plagioclase, 9% chlorite, 8% amphibolite and 4% potassium feldspar. A chemical analysis of the same clay shows a predominance of SiO_2 (53%), followed by Al_2O_3 (17%), Fe_2O_3 (9%), MgO (6%), K_2O (4%), CaO (3%) and Na_2O (2%).

Tiller-Flotten clay is classified as a low plasticity clay and agrees well with the range observed for other Norwegian clays (NGI, 2018) as presented in Figure 1.

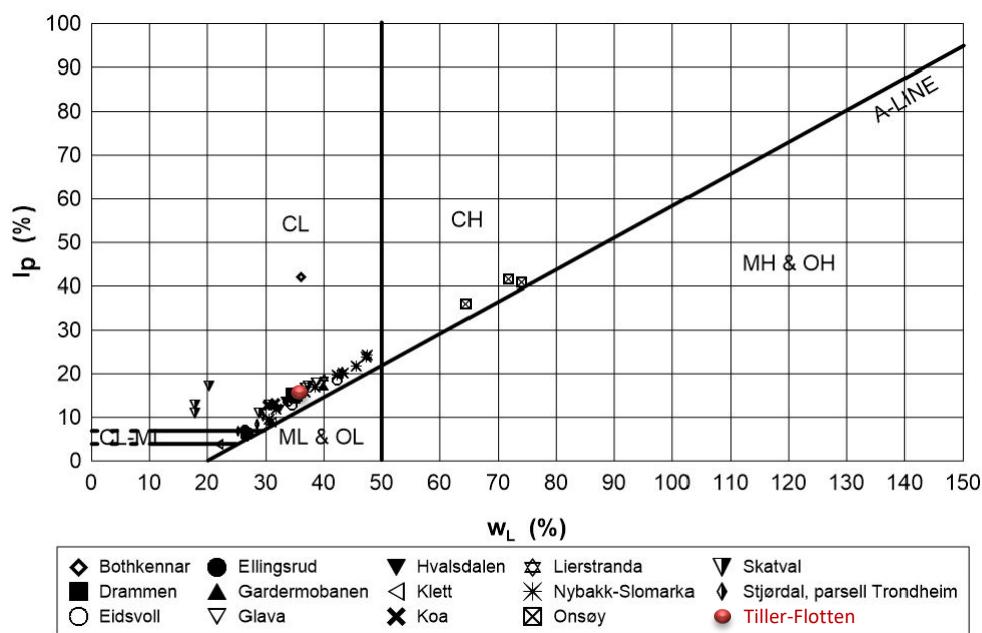


Figure 1 Summary of Atterberg limits of block samples in GEODIP's high quality database (NGI, 2018). The values for Tiller-Flotten are superposed in the figure. CL = low-plasticity clay, CH = high-plasticity clay, ML = low-plasticity silt, MH = high-plasticity silt, OH = high-plasticity organic soil, OL = high-plasticity organic soil

Composition of quick-clays: additional comments

The composition of Norwegian marine clays is (surprisingly) similar. Grain size distribution analysis show commonly higher weight percentage of the silt fraction compared to the clay fraction, and the dominating minerals in the clay fractions are illite and chlorite. The clay deposit at Tiller, Trondheim, is an exception, with clay fractions from 53 to 67% at depths 7,2 to 20,7 m (Hilmo, 1989), indicating that this clay was deposited in a 'brackish water' of Trondheimsfjorden at the time of deglaciation (10800-10000 years BP). Here also, the clay fractions are dominated entirely by illite and chlorite. This is also described by L'Heureux et al. (2019), where the clay content of the quick clay at Tiller-Flotten varies between 70% at 7.5 m to a value of 50% at 19 m, with also biotite, illite and chlorite as dominating minerals.

A large part of the material in the clays are derived from glacial abrasion of the bedrocks, and in Mid-Norway, this includes abrasion of shales, which contain abundant illite, mica (biotite and muscovite), and chlorite. These minerals (larger flat particles) are also found in the silt fractions, having an impact on the grain texture of the clays.

Investigations of boreholes at Dragvoll, Trondheim, some 10 km north of Tiller, show clays with significant lower clay fractions: 33 to 38%. But the dominating clay minerals are also illite and chlorite.

Both clays, Dragvoll and Tiller, are classified as quick clays (remoulded shear strength < 0,5 kPa). The pH is 8,5 and 8,7, respectively. Tiller-Flotten has a pH value of 8,5 (as it will be presented later in this report).

Hilmo (1989) investigated whether there were peptizing substances (i.e. a product that enhances dispersion of a substance, such as clay, into colloidal form) in quick clays, which could explain some of its sensitivity, e.g. silicic acid dissolved from the minerals in the clay system. One of the questions addressed was the effects of pH value. It was concluded that clays with Skempton activity < 0,1, are all characterized by high pH values, from 8,4 to 9,2. The effects of these pH values may be that magnesium- and calcium-ions have precipitated as solid salts in the pore space, thereby reducing the activity of these cations in the pore water. Furthermore, that the high pH values have led to the release of silicic acid from the fine-grained quartz in the clay, and that this dissolved silicic acid may act as a peptizing agent in the clay.

3.4 Laboratory program

The laboratory analyses include UC tests, pH, water content and plasticity measurements performed at NGI laboratory in Oslo. Scanning Electron Microscope (SEM) analyses in selected samples were performed at University of Oslo. XRD and XRF analyses were performed in selected samples at the Norwegian Geological Survey (NGU) in Trondheim.

The clay samples were mixed in the lab with a binder composed of burnt lime product and cement in a 50/50 proportion. The binder content was varied according to Table 2. The samples were cured at room temperature (around 20°C) and tested in unconfined compression (UC) at 28 days curing time. Three samples per mixing were tested. The method for sample preparation follows the one proposed by NGF (2012).

The pH of the mixed samples was measured at 0 (1 hour after mixing), 1, 2, 3, 7, 14, 21 and 28 days of curing.

The plasticity of the mixed materials was measured right after mixing (0 days) and after 24 hours of curing (1 day). The water content was taken at 0, 1 and 28 days of curing.

The same clay type had been previously tested by NGI (2019) with binder contents varying as follows: 30, 60 and 90 kg/m³ for the same binders used in this research. Also, samples with 90 kg/m³ and either 100% cement, 100% LKD or 100% Stabila B100 were prepared and tested in UC. In addition, experiments were performed with 110 kg/m³ for LKD and Stabila B80 to compare the results with NGI (2011). The laboratory results of these tests are presented in NGI (2019) and NGI (2011). They are taken in this technical note for comparison. Table 2 summarizes the laboratory program for the new and the existing data.

Table 2 Laboratory program for testing of binders (new and previous tests)

Binder content [kg/m ³]	Number of UC tests per binder						
	LKD + cement 50/50	B60 + cement 50/50	B80 + cement 50/50	B100 + cement 50/50	B40 100%	B100 100%	Cement 100%
10	-	3	3	3	-	-	-
15	-	3	3	3	-	-	-
20	-	3	3	3	-	-	-
25	-	3 ¹	3 ¹	3 ¹	-	-	-
30	3*	3*, ²	3*, ²	3*, ²	-	-	-
35	3	-	-	-	-	-	-
40	3	-	-	-	-	-	-
45	3	-	-	-	-	-	-
50	3 ¹	-	-	-	-	-	-
60	3*, ²	3*	3*	3*	3*	3*	3*
90	3*	3*	3*	3*	3*	-	-
110	3* & 4**	-	3* & 4**	-	-	-	-

*(NGI 2019)

**Clay from Sognsveien 72. UC performed after 2 and 28 days. Curing temperature was 8°C.

¹One sample analysed by SEM

²One additional sample analysed by XRD and XRF

4 Mechanisms of stabilization with lime and cement

When stabilized with lime and cement, there are several reactions in the soil that contribute to increased strength and stiffness. The chemical process is mainly composed of five reactions: hydration of the binder (dehydration or drying of the clay), ion exchange & flocculation, pozzolanic reactions and carbonation.

The main reaction products are different variants of calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H), which are formed by hydration of cement and by pozzolanic reactions (Bergado et al., 1996). C-S-H and C-A-H are solid crystal phases that harden over time. The crystal phases fill the cavities and forms permanent bonds between the soil particles, which increases the strength in the stabilized masses (Kok & Kassim, 2001)

4.1 Hydration

Cement is a hydraulic binder which mainly consists of calcium silicates and calcium aluminates, that hydrate to form primary-calcium-silicate-hydrate and secondary-calcium-alumina-hydrate, C-S-H and C-A-H. These products contribute to the short- and long-term increase of strength and stiffness. The reaction also produces Ca(OH)₂.

Lime is a binder that does not have hydraulic properties, but which reacts exothermic in contact with water. Burnt unslaked lime is mainly CaO which reacts immediately by reaction with water and forms Ca(OH)₂ (slaked lime) and liberates heat. Ca(OH)₂ dissolves in the pore water and gives an increase in pH in the soil (pH = 12-13).

In both cases, the clay will reduce its pore water content (will dry) and the pH will increase.

4.2 Ion exchange & flocculation

The ion exchange takes places by replacing Ca^{+2} from the lime cement positive ions in the clay minerals, usually Na^+ and K^+ (Prusinski and Bhattacharja, 1999). The addition of calcium ions causes the clay to flocculate and have a coarser and firmer structure (Åhnberg et al., 1995). How much effect this has on the development of strength depends on clay ion exchange capacity, which in turn depends on the pH value, the surface area of the clay minerals and the clay geological origin (Åhnberg et al., 1995; Bergado et al., 1996).

4.3 Pozzolanic reactions

High pH causes silicon and aluminium to be released from the clay minerals. This enables pozzolanic reactions in clay. Pozzolans consist mainly of silicon or aluminium compounds (SiO_2 and Al_2O_3), which are common constituents of clay minerals and quartz (Bergado et al., 1996). In pozzolanic reactions, silicon and aluminium react with calcium hydroxide which to form C-S-H or C-A-H. These reactions lead to the strength and stiffness gradual development (Cherian and Arnepalli, 2015). Pozzolanic reactions are temperature dependent and require also the presence of water.

4.4 Carbonation

Carbonation occurs when clay stabilized material encounters CO_2 in the atmosphere or in the earth. CO_2 decomposes in the pore spaces of clay transforming $\text{Ca}(\text{OH})_2$ or C-S-H into water and calcium carbonate (CaCO_3). This reduces the pH value (Bergado et al., 1996; Xu et al., 2020) and might delay or stop the pozzolanic reactions (Winterkorn & Pamukcu, 1991).

Stabilizing quick clay: additional comments

As described above, there is a set of reactions that help to stabilize the soil. In the case of quick clays, which consist mainly of silt (particle sizes between 2 μm to 63 μm) and a clay fraction (particles $< 2 \mu\text{m}$), there are additional factors contributing to the strength increase. In Central Norway, the content of clay fraction is about 30% by weight +/- 10%. Quick clay from Tiller is exceptional, as it contains between 60 and 70% by weight in the clay fraction. This is of great importance for explaining how the clay is consolidated, for example when drying coarse silt, the main product is a loose powder with almost not binding properties, however, when drying clay, the material become stronger in compression without adding any other material like cement. This is largely due to the attractive van der Waals forces between the clay particles, which come into contact with each other during drying. The addition of cement in wet quick clay is in a sense a drying process, in which the van der Waals forces make a strong contribution.

This should be studied further, since it is out of the scope for this project, by comparing two clays like one with a 30% clay fraction vs. a clay like Tiller-Flotten with ~65% clay fraction.

5 Results

5.1 Water content and plasticity

Figure 2 presents the water content measurements for the samples at 0 days of curing, 1 day of curing and 28 days of curing. The measurement of the water content is according to NS-EN ISO 17892-1:2014. The in-situ water content of the clay is close to 45% at the depth of 9 m (where the samples were taken from). The samples before mixing match the in-situ water content with an uncertainty of $\pm 3\%$. The results show that in general the water content slightly reduces with time from the mixing day to the testing time at 28 days, and the combination of Stabila B100 + cement reduces less the in-situ water content of the clay.

The reduction of the water content indicates the dewatering of the soil ("soil drying") to hydrate the added binders.

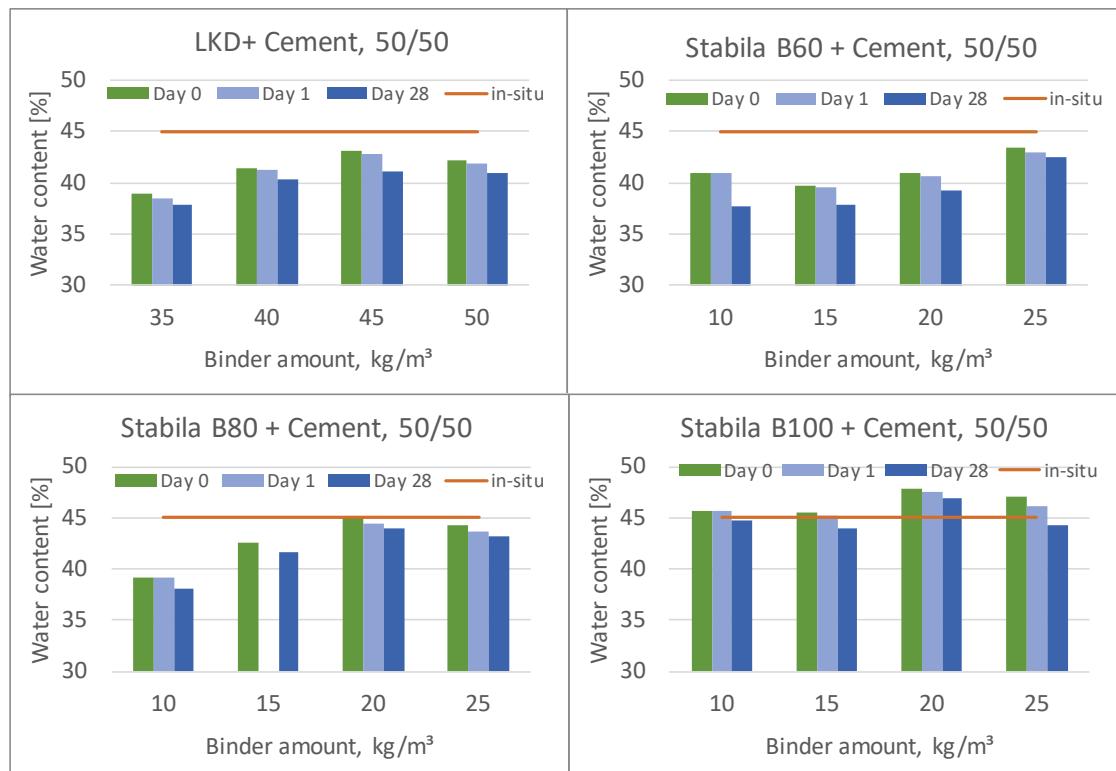


Figure 2 Water content measurements at different curing times. The samples before mixing match the in-situ water content with an uncertainty of $\pm 3\%$.

Figure 3 presents the plasticity measurements (according to NS 8002:1982 and NS 8003:1982) for the samples at 0 days of curing and 1 day of curing. The in-situ plasticity index of the clay is close to 15% at the depth of 9 m (where the samples were taken from). The results show that in general the plasticity slightly reduces after one day of mixing and that the mixed clay has a larger plasticity than the intact clay (the quick clay from Tiller-Flotten is a low plastic clay). The combination of Stabila B100 + cement in the clay gives more stable plasticity values from 0 to 1 day of mixing.

In general, there is an increase in both the plastic and liquid limit (respect to the in-situ state) due to the addition of burnt lime and cement, which also has been observed for Swedish clays (Åhnberg et al. 2003). Then, from day 0 to day 1, the plasticity index reduces slightly, mainly due to the reduction in the liquid limit. The reduction in plasticity after mixing is an indication of the ion exchange (in this case from day 0 to day 1) in the sample reaction, as commented by DiSante et al. (2014) for clayey soils treated with lime.

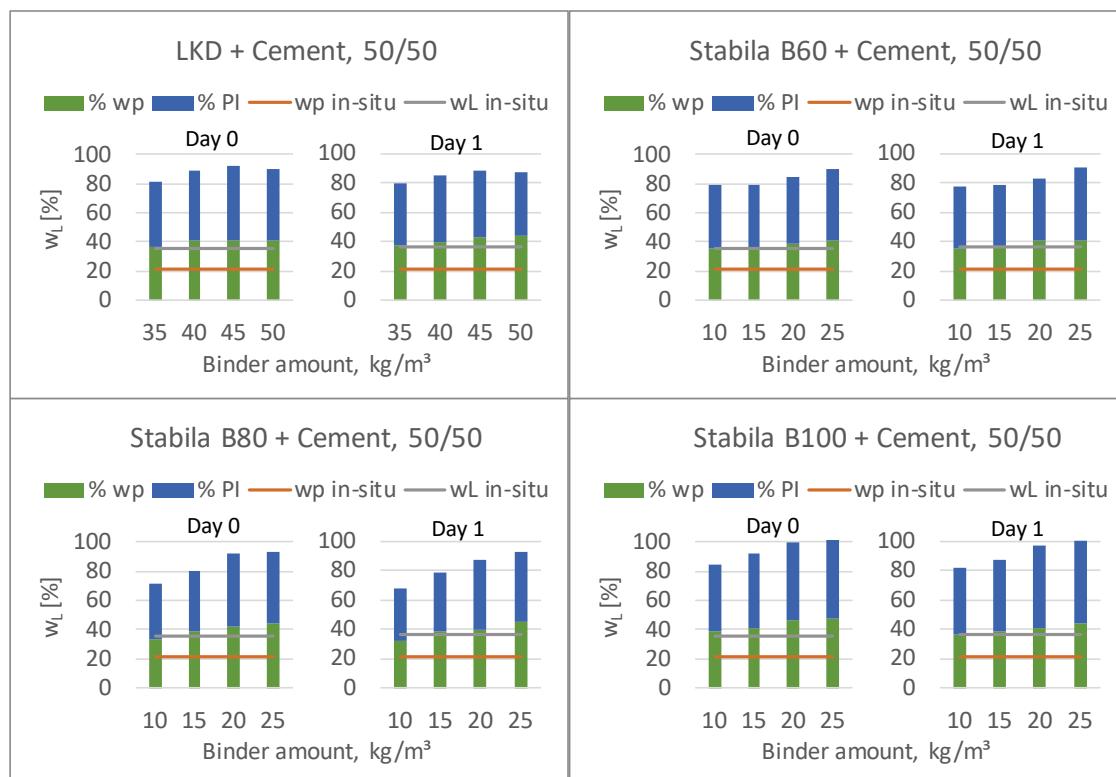


Figure 3 Atterberg limits (w_L : liquid limit, w_p : plastic limit, PI: plasticity index) measurements at two different curing times. In-situ values are shown as a reference.

5.2 pH value

The pH value measurements of the sample mixtures, with curing time from zero to 28 days, are shown in Figure 4. The method for the pH value measurements follows NGI internal instruction based on Houba et al. (1989). The in-situ pH value of the clay used in these experiments is 8,5 and the addition of burnt lime and cement increases the pH value to values between 11.5 to 12.2, depending on the type and amount of cementitious material. Measurements of pH value after 28 days shows values ranging between 10.7 and 11.6, with highest values for the samples with highest contents of binders.

As expected, the pH value measurements are responding very well with the hydrolysis reactions of the added cementing substances (DiSante et al. 2014). The rapid hydrolysis reactions of burnt lime and cement lead to a fast increase of pH value during the first hours, and the temperature of the samples is expected to increase (note that the temperature was not monitored as part of this project). This process also consumes significant amounts of porewater, and calcium hydroxide dissociates in the remaining porewater. This new composition of the porewater is quickly leading to cation exchanges on the clay mineral surfaces, which were initially saturated with mainly sodium and potassium ions in exchangeable positions. Since the divalent calcium ions are balancing more efficiently the electrical charges at the mineral surfaces compared to the monovalent sodium and potassium ions, the electrostatic repulsive forces on the mineral surfaces are also reduced, and the attractive van der Waals forces will dominate. All these reactions contribute to stabilizing the sensitive clay.

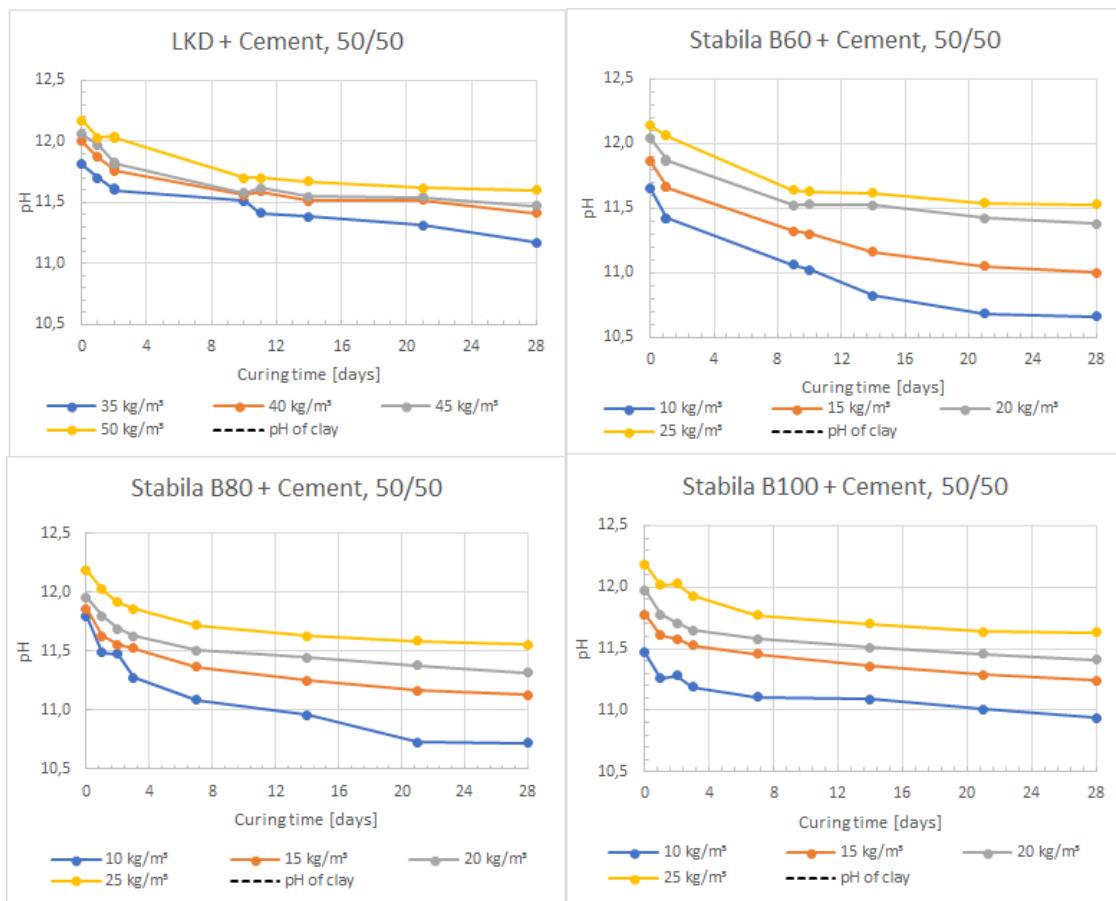


Figure 4 Measurements of pH value at different curing times

When all the cementitious materials have hydrated, and new cement minerals have crystallized, there is less porewater and less dissociated OH- ions in the porewater. At this stage, the pH value of the residual water is expected to stabilize in equilibrium with the new cementing minerals. In addition, the cement used in this project (standard cement FA, CEM II B-M) involves fly ash (pozzolan) which needs an activator (like Ca²⁺) to react as cement minerals do, this contributes to the reduction in Ca(OH)₂ and therefore in pH value. Excess Ca(OH)₂ will give an equilibrium pH value between 11 and 12, as seen on samples with high contents of burnt lime and cement (e.g. pH value 11,7), while other minerals, like calcium silicates and calcium-aluminium-silicates, will have lower pH value of equilibrium. Marine clays commonly contain some calcite (shell materials), which give an equilibrium pH value close to 10. Similar pH value is expected with non-calcinated lime in e.g. LKD.

One should also bear in mind that marine clays contain significant amounts of fine-grained quartz. Quartz is a very stable mineral at ambient conditions, but solubility of quartz in water increases exponentially with increasing temperature and pH value (above pH value 9). It is therefore expected that some dissolved quartz contributes to the formation of calcium-silicate during the curing of lime/cement stabilized clay.

5.3 Strength

Figure 5 presents the measurements of strength from unconfined compression (UC) tests of stabilized clay samples at 28 days of curing time, for different binder contents and binder types. The method follows NS-EN ISO 17892-7:2017.

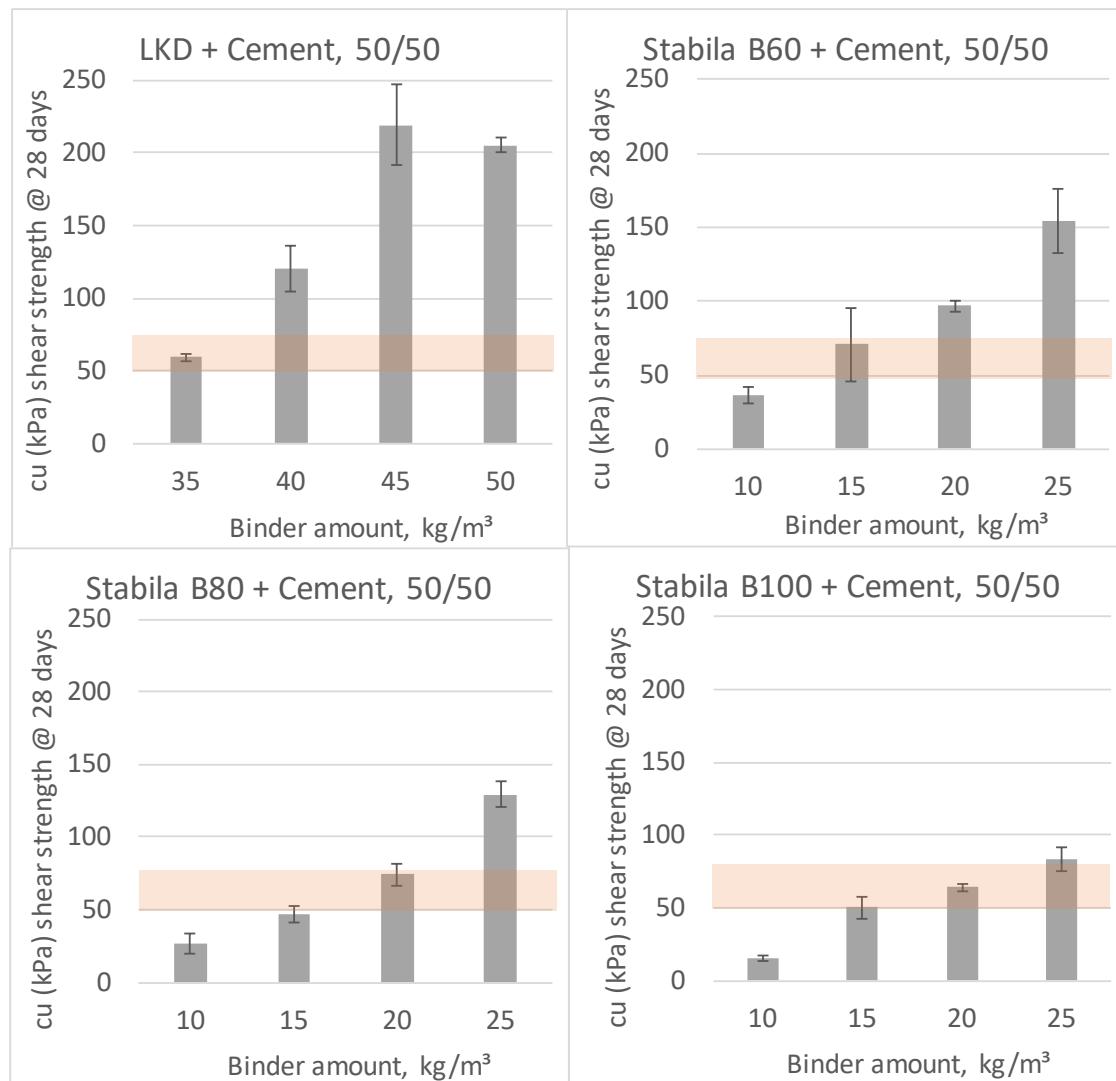


Figure 5 Average strength from unconfined compression tests of stabilized clay samples at 28 days of curing time. The line bars in each column indicates the standard deviation.

The binder content for LKD + cement is larger than the binder content used for the other binder types, which also reflected in the magnitude of strength values reached. The in-situ strength of the clay from UC tests varies between 50-75 kPa at 9 m depth (area marked in orange in the plots). The samples with the largest binder content (i.e. 25 kg/m³) of Stabila B60 + cement and Stabila B80 + cement reach strength values over the in-situ. This behaviour is not observed for Stabila B100 + cement with the same

binder content. This might be due to insufficient hydration of the binder, as the water content measurements show (Figure 3); however, this needs to be further studied by also adding temperature measurements during curing time, for example. This comparison is done in general to show the capabilities of the binder mixtures to improve the in-situ strength and stiffness of the clay, without accounting for the curing stress conditions (i.e. unconfined conditions in the lab vs. in-situ stress in the field).

Appendix B presents all the data from the laboratory results.

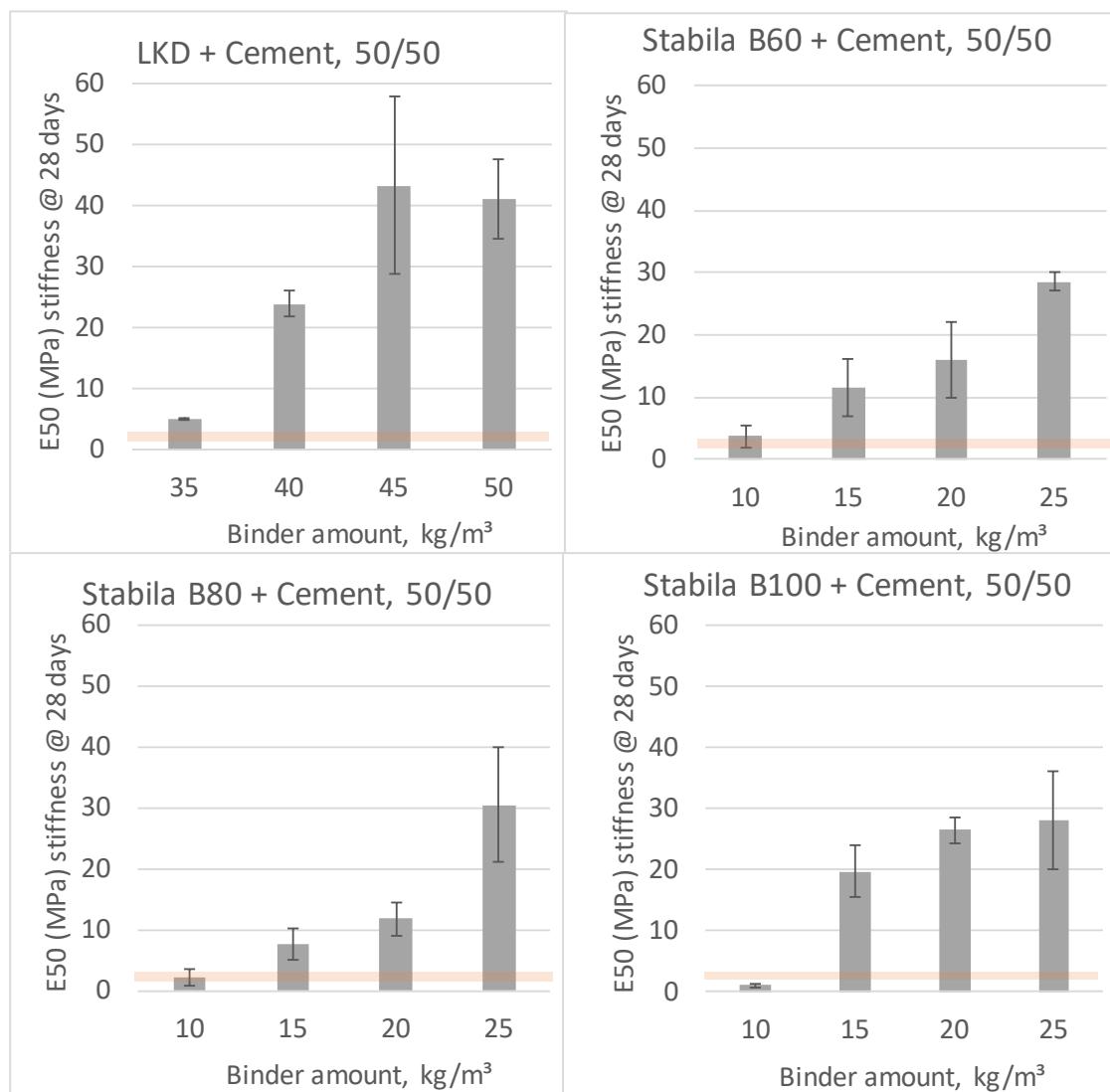


Figure 6 Average stiffness E_{50} from unconfined compression tests of stabilized clay samples at 28 days of curing time. The line bars in each column indicates the standard deviation.

5.4 Stiffness

Figure 6 presents the measurements of stiffness from unconfined compression (UC) tests of stabilized clay samples at 28 days of curing time, for different binder contents and binder types. The binder content for LKD + cement is larger than the binder content used for the other binder types, which also reflected in the magnitude of stiffness values reached. The in-situ stiffness (E_{50}) of the clay from UC tests is around 2,5 MPa. An increase in the stiffness from the in-situ value is observed from a binder content near 15 kg/m³ for all types of binder, except for LKD + cement where this increase is observed at 40 kg/m³.

Appendix B presents all the data from the laboratory results.

5.5 SEM investigations

Appendix C presents the results of a scanning electron microscope (SEM) analyses performed at the University of Oslo with the objective of obtaining high-magnification imaging and mineral chemistry. Four samples were tested in SEM with 50 kg/m³ for LKD + cement and 25 kg/m³ for the rest of binder types. All binders were added in a 50/50 ratio. The samples were 98-102 days old (~3 months) from the mixing day.

The images for samples stabilized with B40 + cement show needle structures that may indicate products of the pozzolanic reactions. These structures were not observed in the other samples tested. This might be due to the age of the samples since at the time of SEM imaging, the cementitious reactions might have stopped for these samples.

Further analysis of SEM images

In standard cement FA is commonly added 2-5% gypsum as a retarder. During the initial hydration of the cement (4 to 6 hours), this gypsum binds the tricalcium aluminates (C₃A) to form ettringite. The SEM micrographs in Appendix C, Figures 2A and 2B show these needle shaped ettringite crystals. This is indicated by higher sulphur values on EDS data. The patchy distribution of ettringite crystals in the sample is probably due to the initial distribution of gypsum particles.

The longer ettringite crystals bind the cement and clay particles together. During the further hydration of the clinker materials, long intertwined fibres of calcium silicate hydrate crystals are formed (mainly from tricalcium silicates, C₃S) which further consolidate the particle structure.

After one day of hardening, the interstices of the clay-cement mixture are filled with various hydration products and the strength is further increased. These new products of hydration are difficult to identify on EDS since their composition is similar to the background.

5.6 XRD & XRF investigations

The XRD and XRF analyses (in bulk samples) of stabilized clay samples were performed at NGU at 28 days of curing. The complete analysis report is presented in Appendix D. Four new samples were mixed for these analyses:

- ↗ Stabila B40 + Cement, binder content = 60 kg/m³
- ↗ Stabila B60 + Cement, binder content = 30 kg/m³
- ↗ Stabila B80 + Cement, binder content = 30 kg/m³
- ↗ Stabila B100 + Cement, binder content = 30 kg/m³

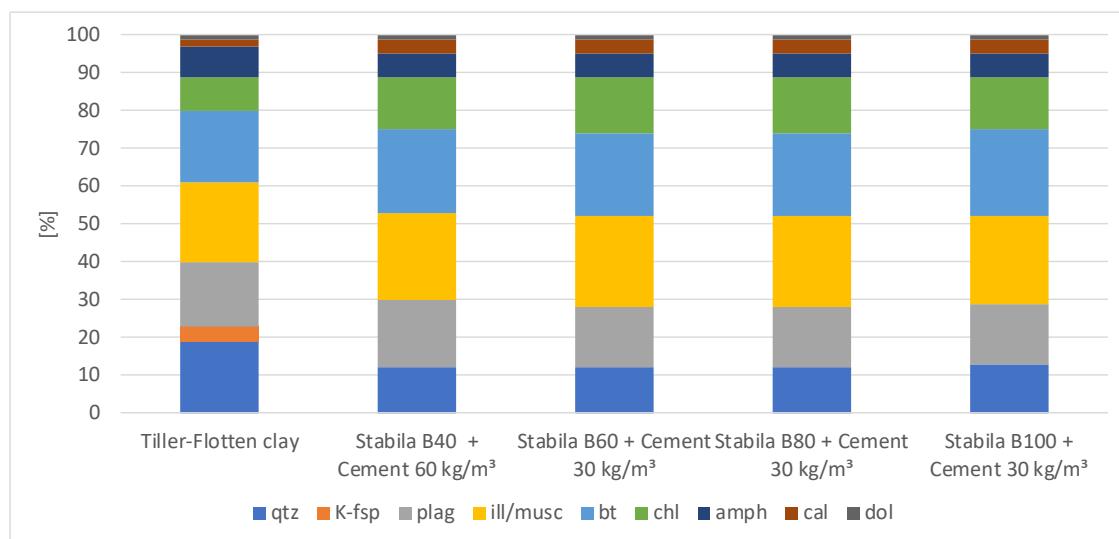


Figure 7 Results of the XRD analyses on selected samples of stabilized clays. The composition of Tiller-Flotten clay is presented as a reference.

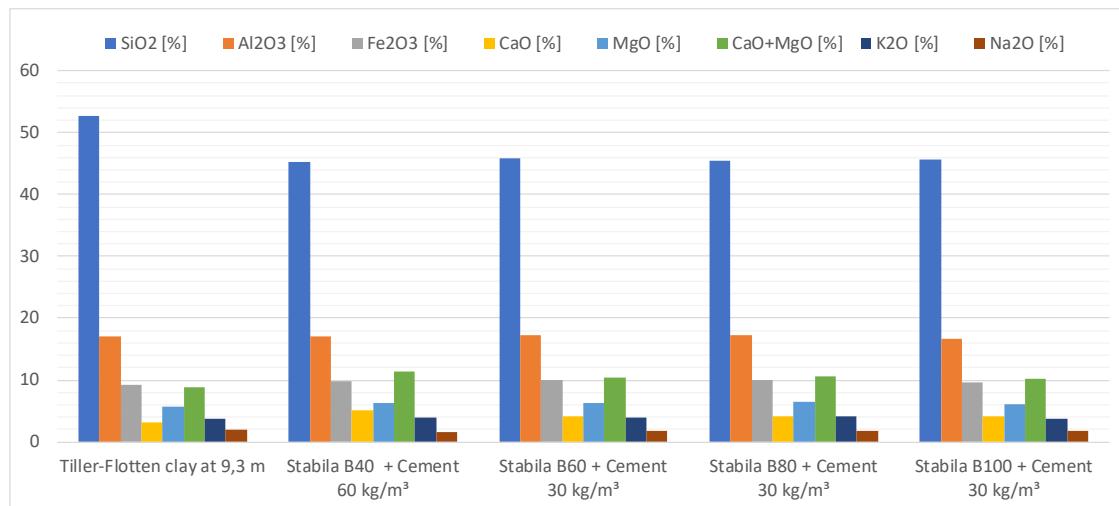


Figure 8 Results of the XRF analyses on selected samples of stabilized clays. The composition of Tiller-Flotten clay is presented as a reference.

The results for XRD and XRF analyses are presented in Figure 7 and Figure 8, respectively. The XRD results show a lower content of quartz and no presence of potassium feldspar in the stabilized clay respect to the original clay material. There is also an increase on calcite in the stabilized clay respect to the original clay. There are not significant differences in the mineral composition of the stabilized clays with the different burn lime types.

The XRF results show that the SiO₂ reduces in the stabilized clay compared to the original clay material and that this reduction is similar for all types of cementitious materials added. The CaO increases in the stabilized clay compared to the original clay materials.

6 Comparison with previous data

The data obtained in the present study has been compared to the data obtained by NGI (2019). Figure 9 and Figure 10 present the data from unconfined compression tests of stabilized clay samples at 28 days of curing time. The highest strength values (i.e. 700 kPa) is reached with Stabila B80 + cement and 110 kg/m³, followed by 500 kPa for all binder types added as 90 kg/m³ (excepting LKD and B100, in 100% proportion, which reached 250 kg/m³). Strength values over 300 kPa are obtained for 60 kg/m³ of LKD + cement and 30 kg/m³ for Stabila B60 + cement, Stabila B80 + cement and Stabila B100 + cement.

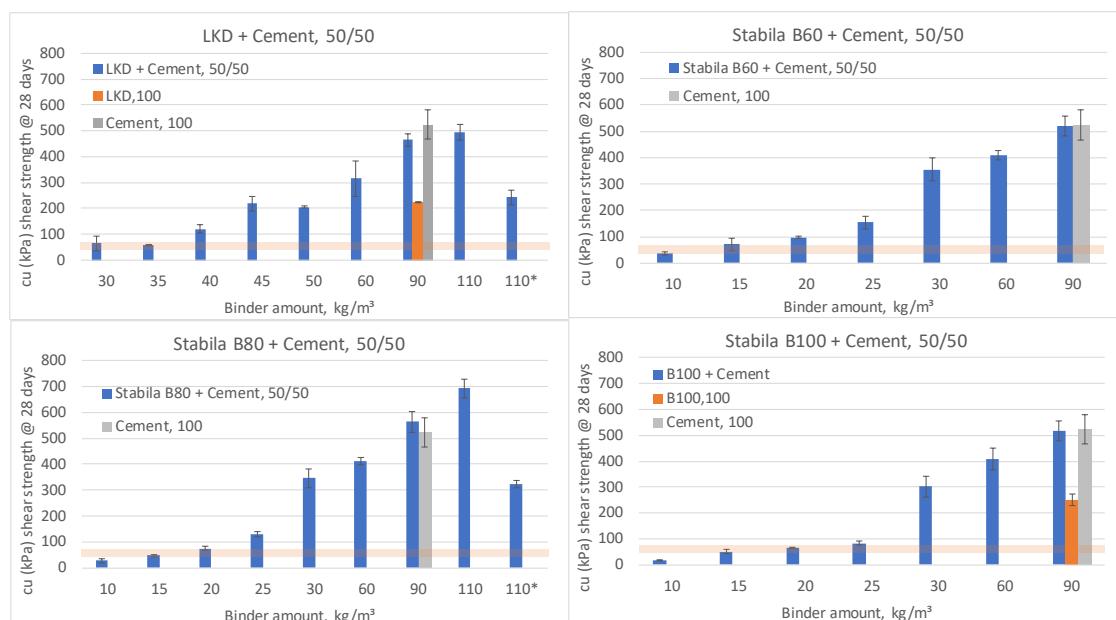


Figure 9 Strength from unconfined compression tests of stabilized clay samples at 28 days of curing time: data from SUSI and data from NGI (2019). The values marked with * were cured at 8 °C.

Figure 9 and Figure 10 also show that the shear strength increases with increasing binder content. However, the increase is not linear, so that a doubling or tripling of the binder content from 30 kg/m³ to 60- or 90 kg/m³ does not give a doubling or tripling in the strength. At the same time, there is a clear increase in strength with an increased proportion of burnt lime in the binder, from LKD to Stabila B60. From Stabila B60 up to Stabila B80, the difference is not large, but here the difference in the proportion of CaO-active is also small. The reason why the strength does not increase further is unknown but might be due to a lack of access to water in the stabilized material, so that the binder does not react completely.

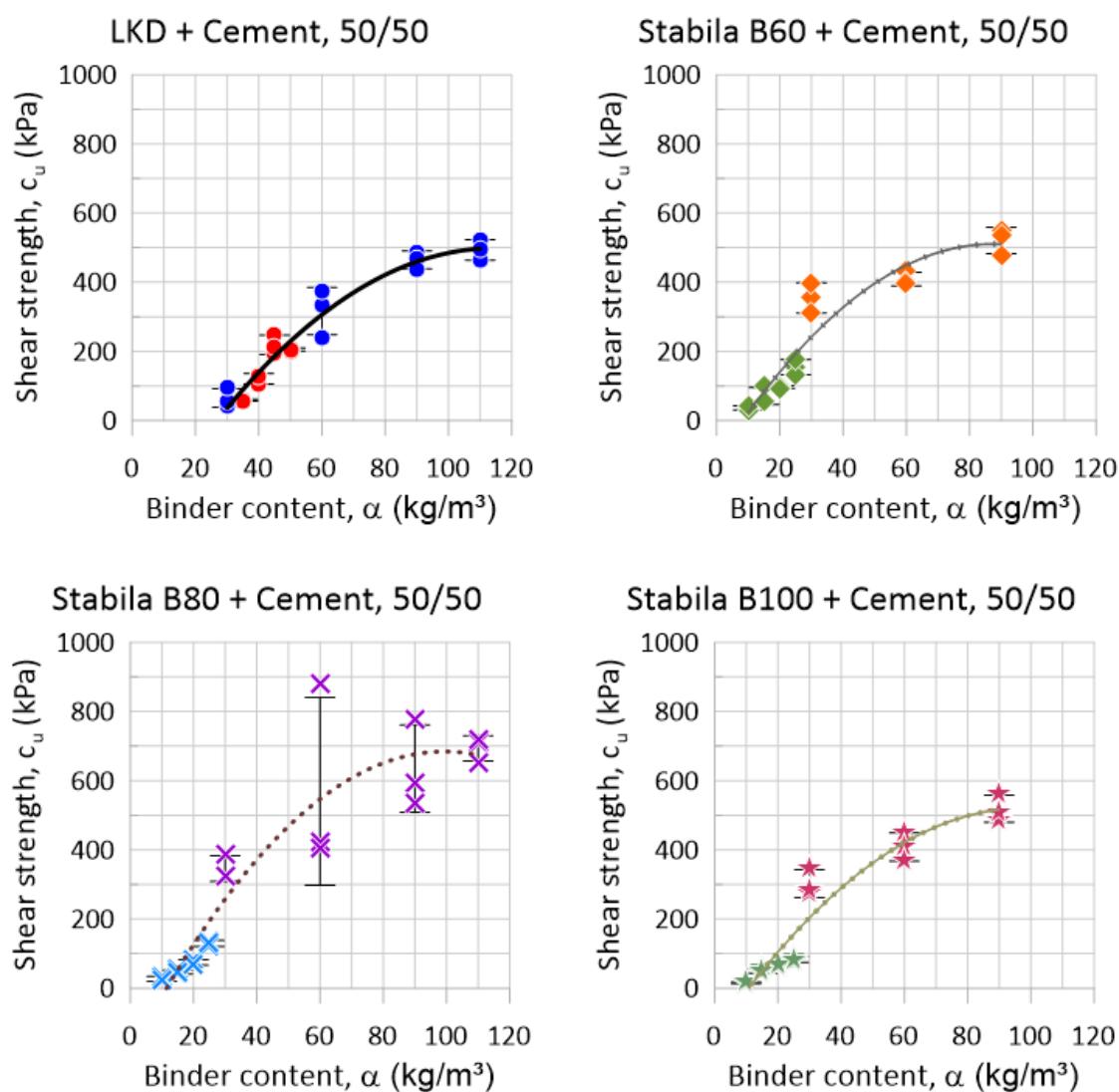


Figure 10 Strength from unconfined compression tests of stabilized clay samples at 28 days of curing time: data from SUSI and data from NGI (2019).

It is possible to identify (Figure 10) that the trend of the data seems to plot in a S-shape curve, where the points of maximum curvature may indicate the minimum binder content necessary to improve strength and deformation properties of this sensitive clay (see Table 3). This trend is clearer when plotting the average values (Figure 11a) and adding a proportional correction for binder content due to the amount of CaO-active (assuming that the cement has 100% activity), see the (Figure 11b).

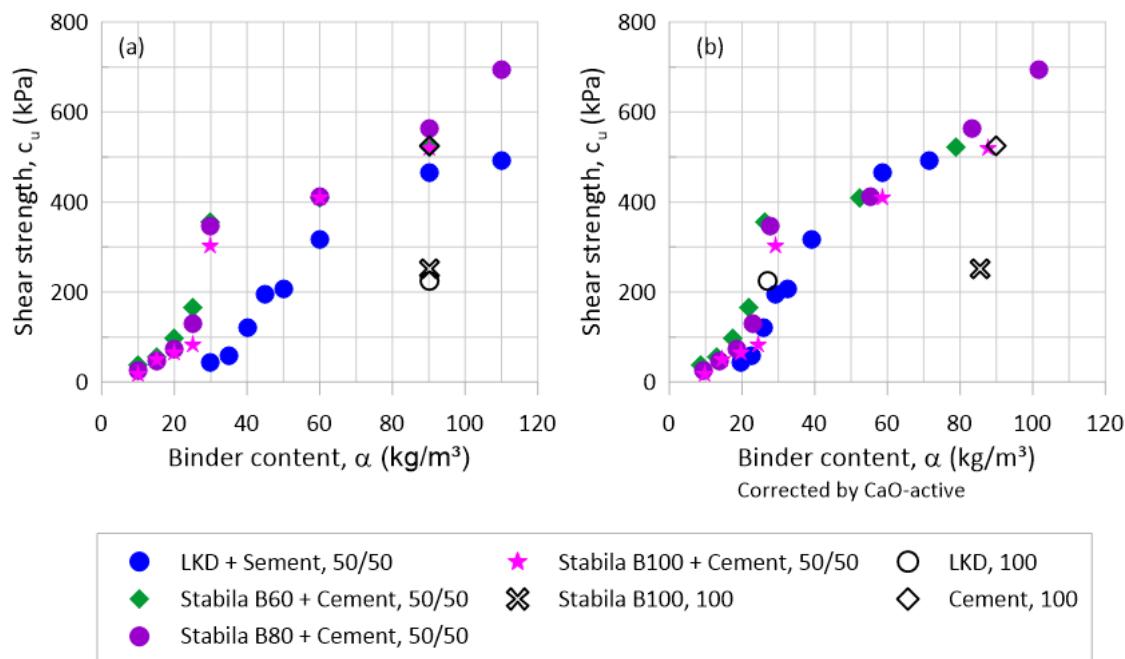


Figure 11 Average strength from unconfined compression tests of stabilized clay samples at 28 days of curing time: data from SUSI and data from NGI (2019).

Table 3 Optimal values of binder content necessary to improve strength and deformation properties of Tiller-Flotten quick clay.

Binder type, 50/50	Binder content (kg/m^3)	Reached shear strength at 28 days (kPa)
LKD + cement	60	300
Stabila B60 + cement	30	300
Stabila B80 + cement	30	300
Stabila B100 + cement	30	300

The values from Table 3 are easily identified when looking at number of CO₂-equivalents from production divided by the strength achieved (Figure 10). This also gives an indication of the climate effect of the different binder types. A minimum value is reached for 60 kg/m^3 of LKD + cement and for 30 kg/m^3 of the other binder types. The results indicate the beneficial climate effect when using a binder with a low proportion of burnt lime (CaO) and a reduced binder content.

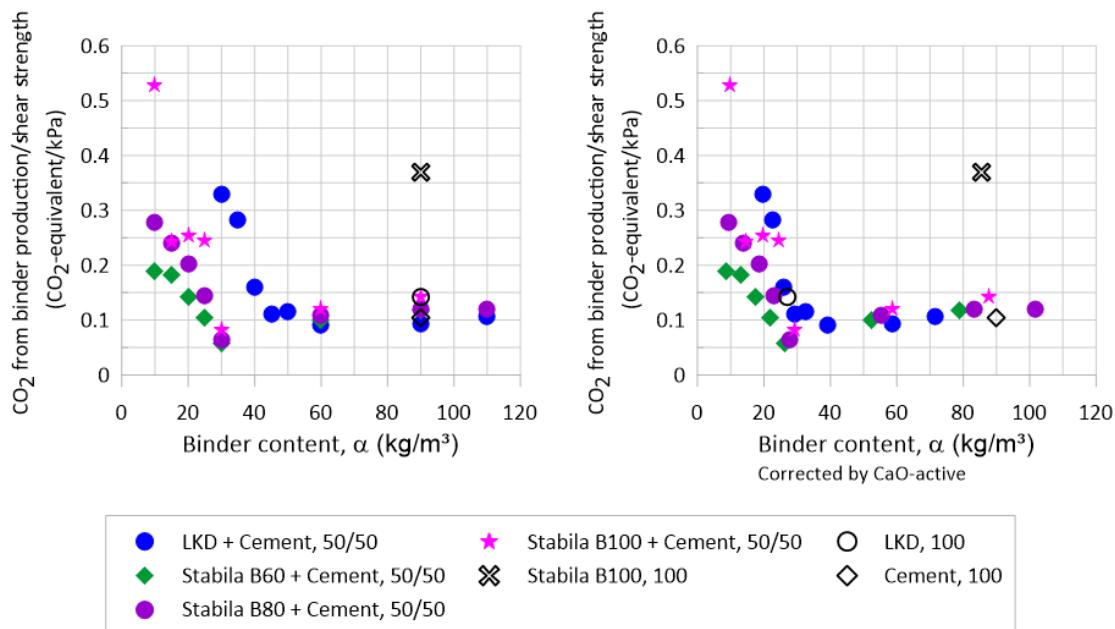


Figure 12 Number of CO₂-equivalents from production normalized by the average strength from unconfined compression tests of stabilized clay samples at 28 days of curing time: data from SUSI and data from NGI (2019).

Regarding the stiffness values (E_{50}), Figure 13 shows that stiffness values over 100 MPa are reached for samples stabilized with 100% cement added as 90 kg/m³, LKD + cement added as 90 kg/m³, Stabila B60 + cement and Stabila B80 + cement both added as 30 kg/m³. Samples stabilized with Stabila B100 + cement tend to have a stiffer response after 30 kg/m³ of binder content without reaching 80 MPa. A large stiffer response is observed at 60 kg/m³ for samples stabilized with LKD + cement.

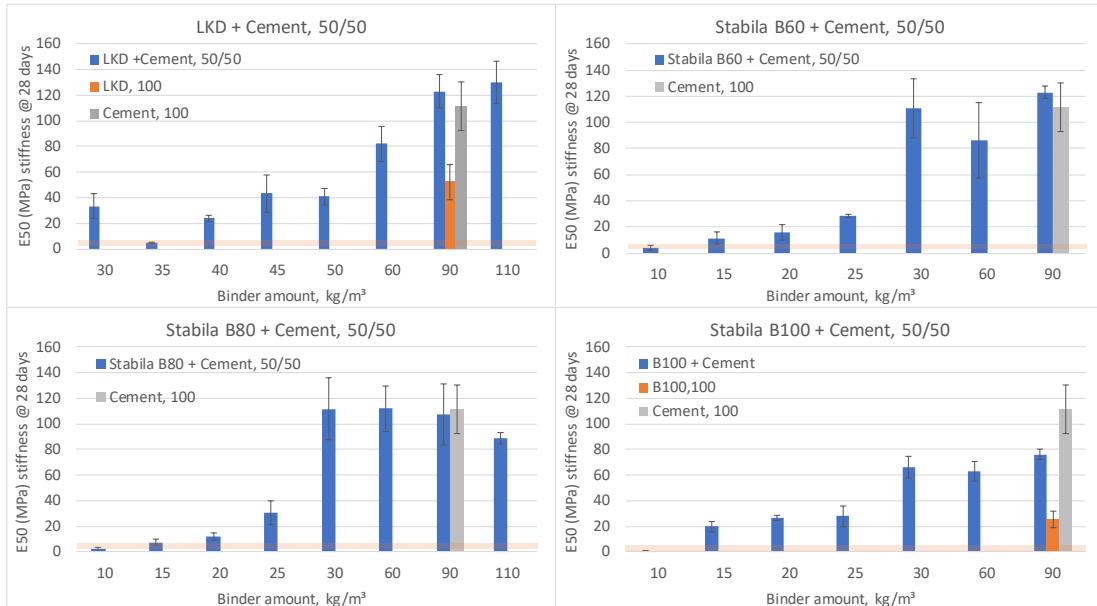


Figure 13 Average stiffness E_{50} from unconfined compression tests of stabilized clay samples at 28 days of curing time: data from SUSI and data from NGI (2019).

7 Conclusions

Based on the results, the following conclusions can be drawn:

- ↗ The water content of the clay reduces when adding cementitious binders. The reduction continues with time. The reduction of the water content indicates the dewatering of the soil ("soil drying") to hydrate the added binders.
- ↗ The plastic and liquid limit increases due to the addition of cementitious binders. Then, with the time, the plasticity index reduces slightly, mainly due to the reduction in the liquid limit.
- ↗ The pH value of the clay increases when adding cementitious binders and then reduces with time.
- ↗ Stabilized clay shows a reduction in quartz and SiO₂, and an increase in CaO from the natural state.
- ↗ A change in the curing temperature from 8°C to 20°C gives an increase in the strength of around 50%, for binder contents of 110 kg/m³.
- ↗ Based on SUSI and previous results, the optimum amount of binder for increase in strength and stiffness is 60 kg/m³ for low reactivity binders (LKD + cement) and 30 kg/m³ for higher reactivity binders (B60 + cement, B80 + cement, B100 + cement).
- ↗ Measurements of water content, Atterberg limits (plasticity) and pH value tend to stabilize at similar values near the optimum amount of binder mention in the previous point.
- ↗ The optimum binder contents give a strength increase (in unconfined conditions) up to 300 kPa. These optimum values for binder content correspond also to the minimum number of CO₂-equivalents from binder production.
- ↗ There is a beneficial climate effect when using a binder with a low proportion of burnt lime (CaO) and a reduced binder content

8 Acknowledgments

To RFF Trøndelag (Regionale forskningsfond Trøndelag) for their generous grant 310057 to perform the project Sustainable Soil Improvement (SUSI).

To the partners in SUSI: Martin Mengede (Franzefoss Minerals), Håkon Rueslåtten (JLE Grunnforsterkning AS) and Caroline Mevik (Melhus municipality) for valuable comments in the present technical note.

To the project Norwegian GeoTest Sites infrastructure (NGTS), grant No. 245650/F50 from the Research Council of Norway, for facilitating the clay materials and the data that describes it.

A special thanks to NGI colleagues: Sølve Hov, Bjørn Kristian Bache, Christian Sætre, Thomas Vestgården and Vidar Gjelsvik who significantly contributed to the work presented in this technical note.

9 References

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Appendix A

DESCRIPTION OF LKD, STABILA B60, STABILA B80 AND STABILA B100



Analyserapport

AR-FMI-19-0036

Produkt:	Stabiliseringssprodukter				Stabila B40 Stabila B60 Stabila B80 Stabila B100	Franzefoss Minerals AS Olav Ingstads vei 5 NO-1309 Rud Telefon: +47 05255 E-post: post@kalk.no
Metode:	Kjemi	WD-XRF	ASTM-E1621-13	CaO-aktiv	ASTM C25	FRANZEF OSS MINERALS
	Reaktivitet Mineralogi	t 60, ΔT Rietveld	NS-EN 459	ekstern		
Metode	Parameter	Enhet	Stabila B			
			40	60	80	100
ASTM-E1621-13	CaO + MgO	[%]	>70	>90	<96	>96
ASTM C25	CaO-aktiv (a)	[%]	<30	75±5	85±5	95±5
Beregnet	CaCO ₃ ber. fra CO ₂ -rest (b)	[%]	>70	20±5	10±5	<3
	Σ Kalk (a+b)	[%]	100	100	100	100
NS-EN 459	t 60	[sek]	n.a.	>500	>180	<180
	ΔdT	[%]	<20	>40	<50	>50
Rietveld	Brentkalk	CaO	[%]	-	-	>96
	Kalsitt	CaCO ₃	[%]	-	-	<3
Stabila B produkter: Kjemi, Reaktivitetskurver, Partikkelfordeling						
<p>Innhold [%]</p> <p>Stabila B</p> <ul style="list-style-type: none"> CaO CaO-aktiv (a) CaCO₃ (b) 						
<p>Temperatur [°C]</p> <p>Tid [sek]</p> <ul style="list-style-type: none"> Stabila B100 Stabila B80 Stabila B60 Stabila B40 						
<p>Gennomgang [%]</p> <p>Størrelse [µm]</p> <ul style="list-style-type: none"> Stabila B100 Stabila B80 Stabila B60 Stabila B40 						
				06.09.19	Martin Mengede	
				Dato	Godkjent	

Analyseresultatene gjelder kun denne analyserapporten og er bare gyldig ved godkjenning. Kopiering av analysen må godkjennes.

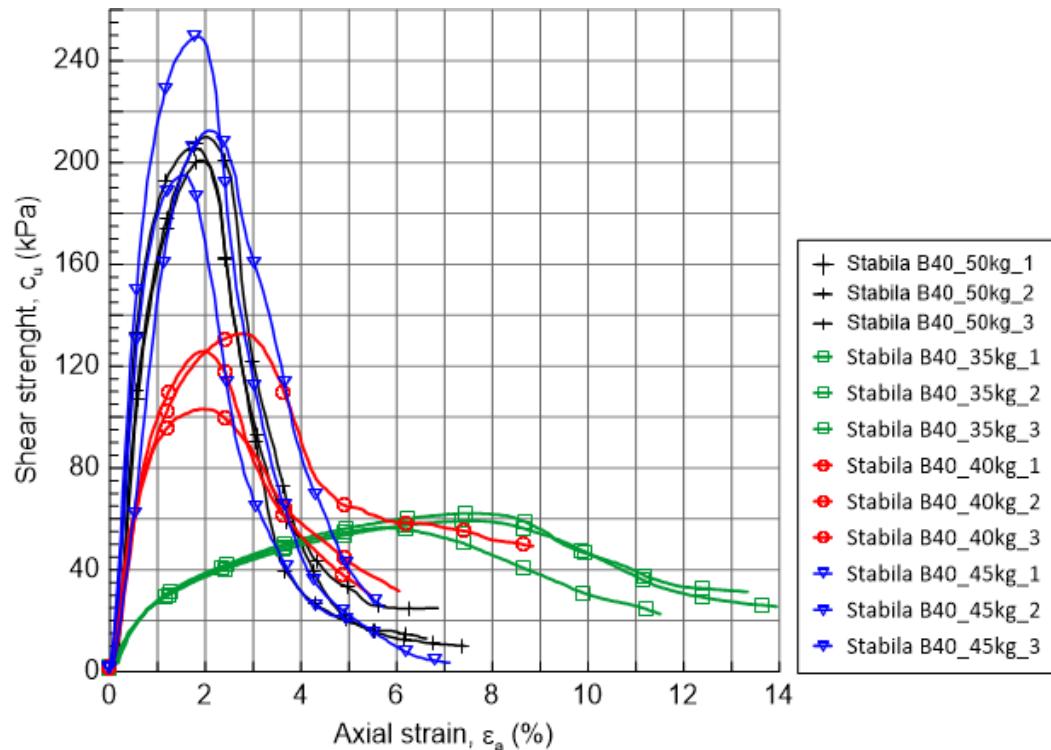
Appendix B

LABORATORY RESULTS

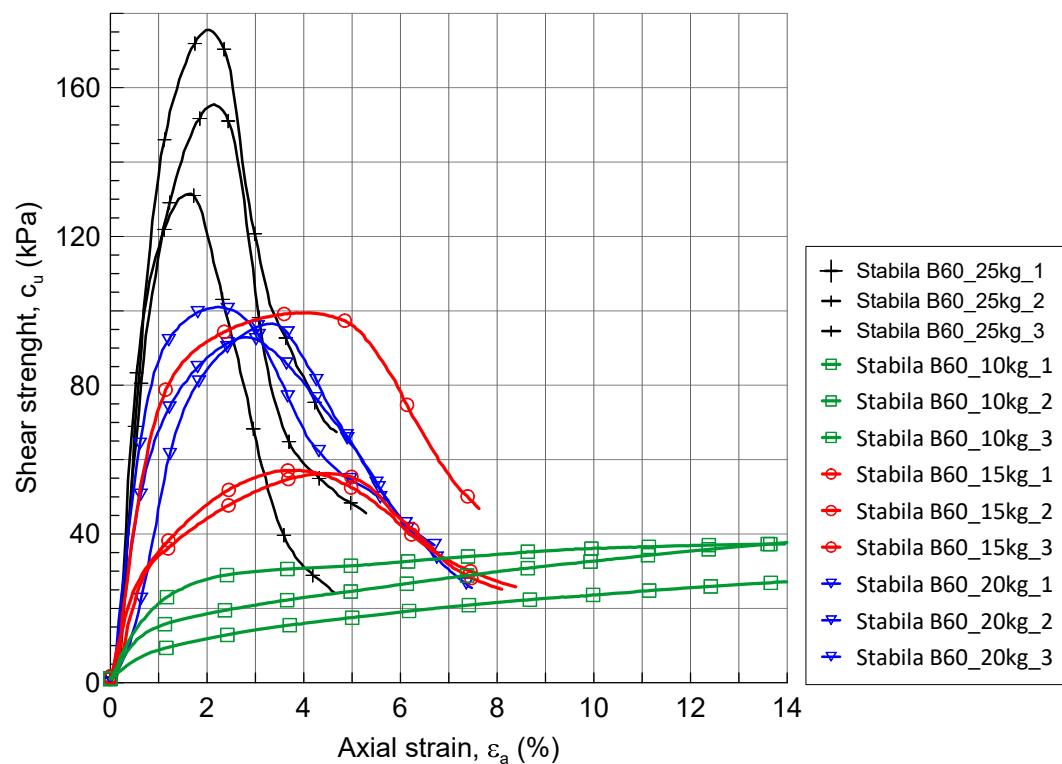
Contents

B1 Stabila B40 + Cement	2
B2 Stabila B60m + Cement	2
B3 Stabila B80m + Cement	3
B4 Stabila B100m + Cement	3

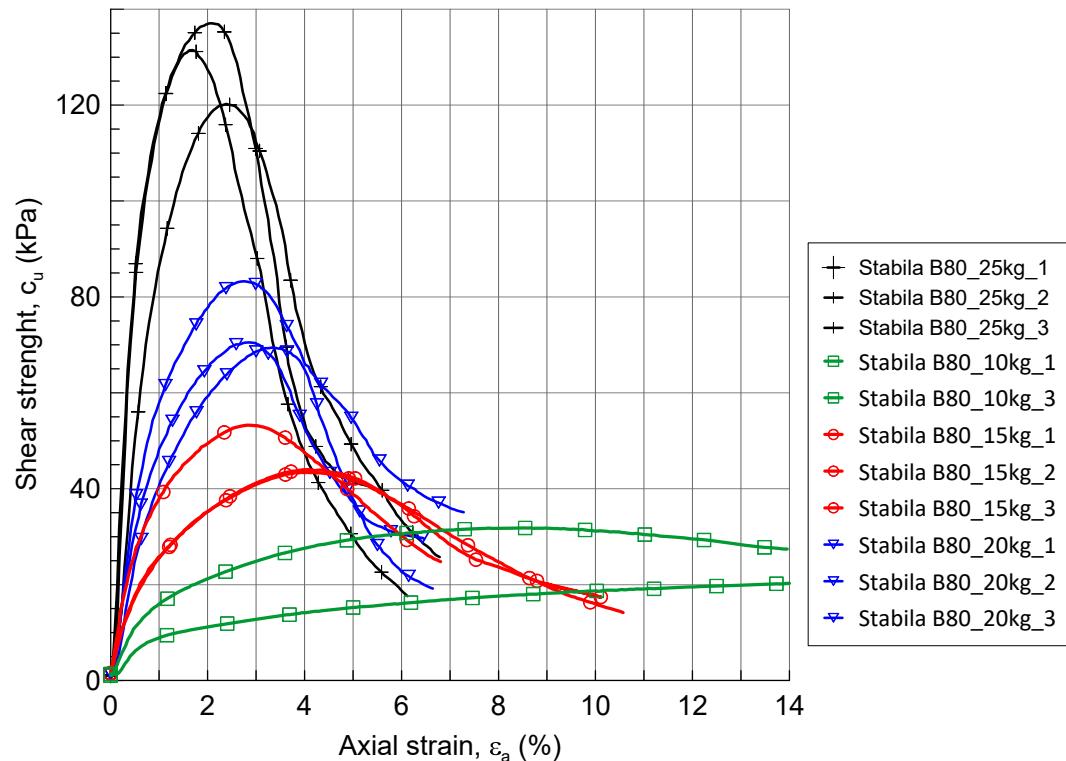
B1 Stabila B40 + Cement



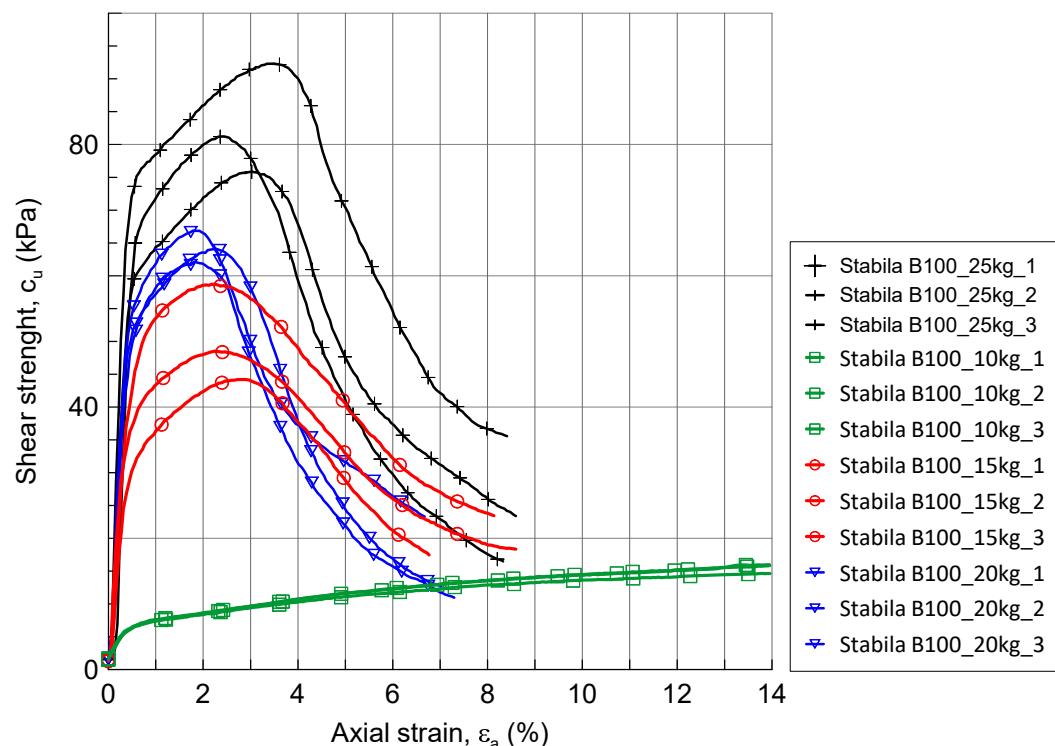
B2 Stabila B60m + Cement



B3 Stabila B80m + Cement



B4 Stabila B100m + Cement



Appendix C

SEM ANALYSIS

Contents

C1 Method	2
C2 LKD + cement (B40)	2
C3 Stabila B60 + cement (B60), Stabila B80 + cement (B80) & Stabila B100 + cement (B100)	5

C1 Method

A scanning electron microscope (SEM) at the University of Oslo was used to obtain high-magnification imaging and mineral chemistry. Samples were carbon coated and analysed with a Hitachi SU5000 FE-SEM scanning electron microscope. Images were acquired using detectors for secondary-electron images (SEI), backscattered electron images (BSE) and ultra-variable detector (UVD). Mineral chemistry analysis was performed with a Dual Bruker XFlash30 Energy Dispersive X-ray Spectroscopy (EDS). The samples were 98-102 days old (~3 months) from the mixing day.

C2 LKD + cement (B40)

Figure 1 shows an overview image of the B40 sample. In the image clay minerals are observed as the ground mass of the sample where larger minerals are embedded. The larger mineral grains appear detrital due to common rounding and lack of minerals showing euhedral shapes. The sample does not show any preferred mineral orientations.

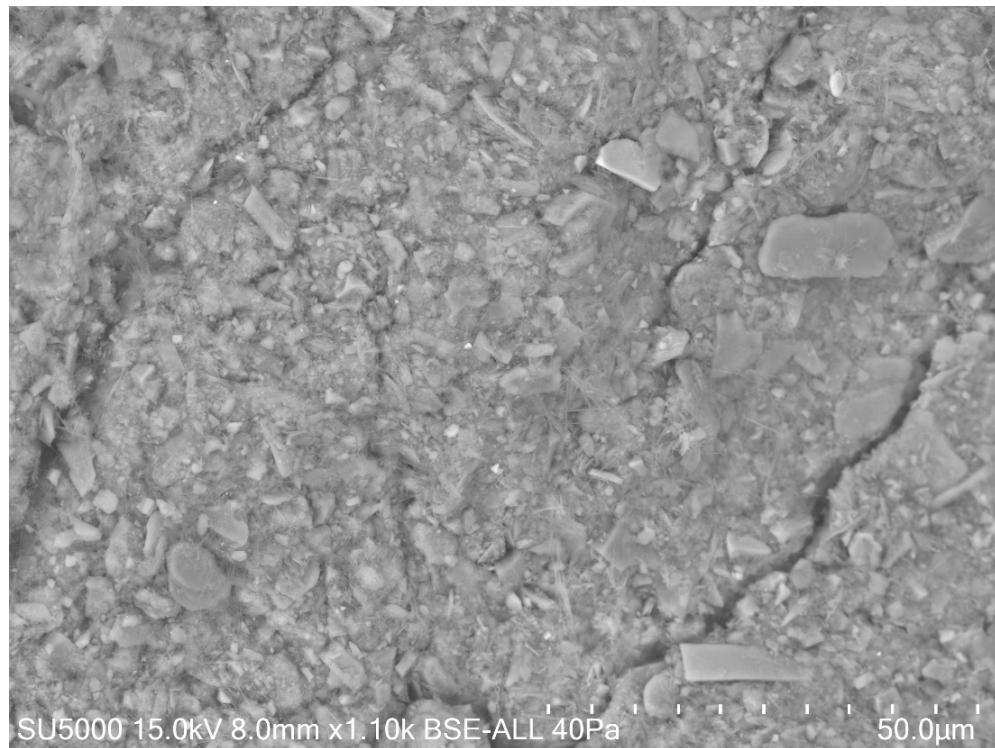


Figure 1 Overview image (BSE) of the B40 sample.

In some locations, small minerals (maximum size 10 µm) showing a needle like form can be observed (Figure 2A and B). They are present with a patchy occurrence in sample B40. The appearance of the mineral suggests a secondary origin. EDS measurements of the needle like minerals are difficult due to a large analytical volume, but they have slightly elevated sulphite (S) and calcium (Ca) measurements indicating possible gypsum or anhydrite.

The SEM-EDS analysis do not show any strong evidences for secondary minerals besides the abovementioned needle like minerals.

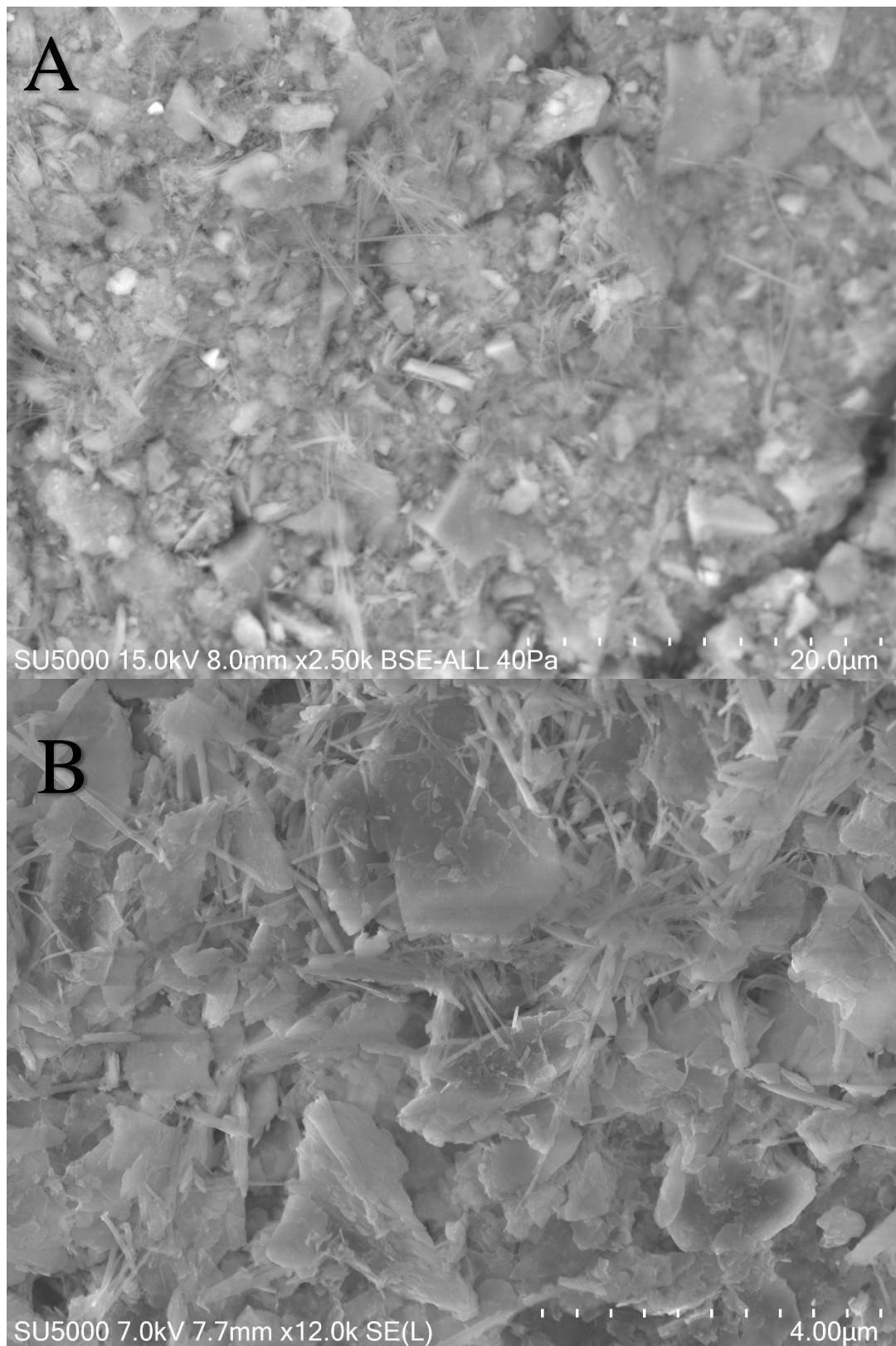


Figure 2 A. Overview of possible secondary minerals with a needle structure. B. close-up image of the same minerals in A.

C3 Stabila B60 + cement (B60), Stabila B80 + cement (B80) & Stabila B100 + cement (B100)

Samples from B60, B80 and B100 are described together as they appear similar under SEM analysis. All samples contain a significant amount of clay minerals where larger grains are embedded (Figure 3 A, B and C). EDS analysis of the larger grains show typically quartz and feldspars, these minerals appear detrital.

There are some minerals, based on appearance, that may be secondary (Figure 4). These are most prominent in the B60 and B80 samples. It is not possible to identify the mineral species based on appearance alone, EDS analysis of the minerals are not available.



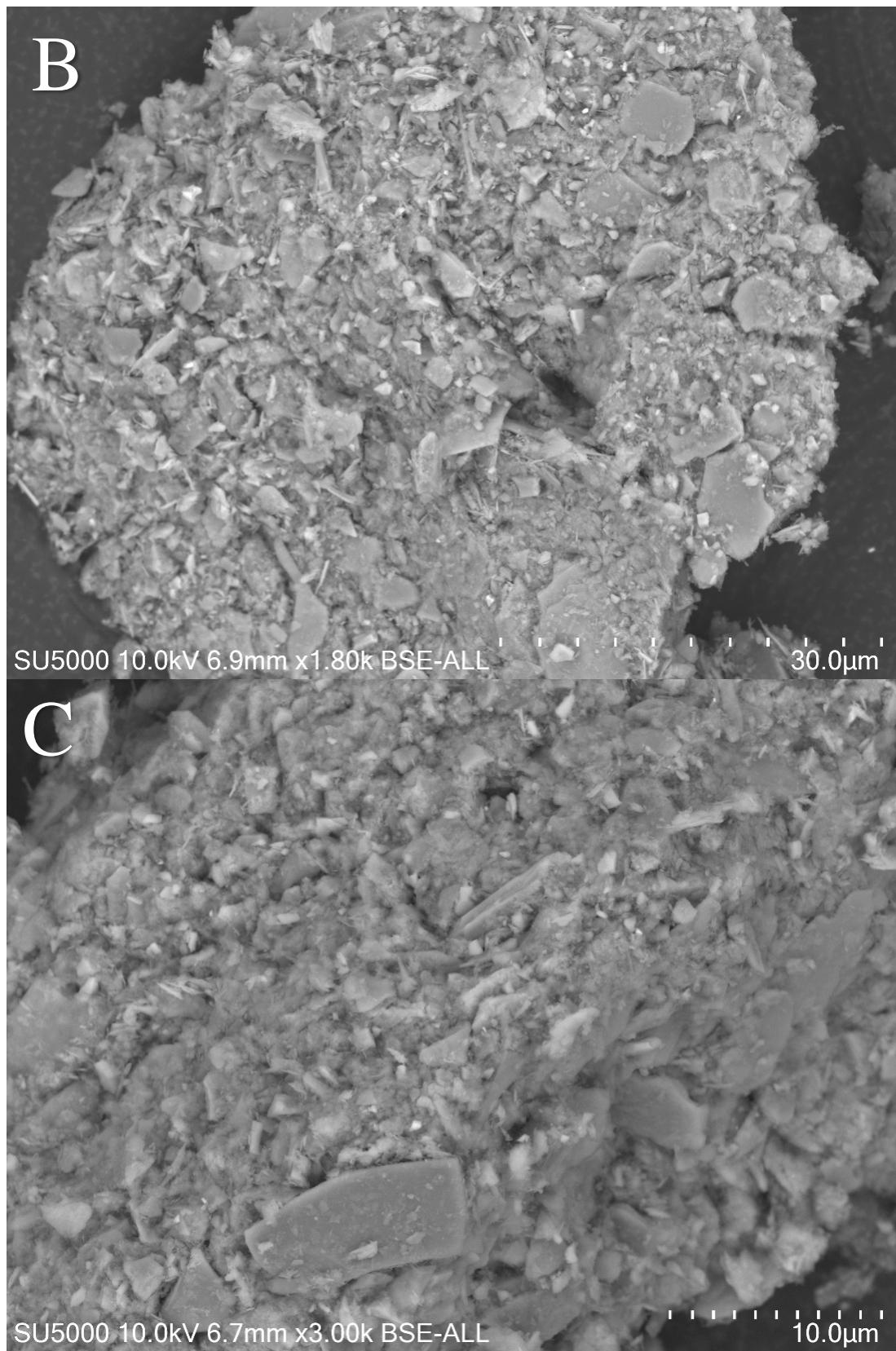


Figure 3 Overview images of: A. B60, B. B80 and C. B100. Note difference in scale.

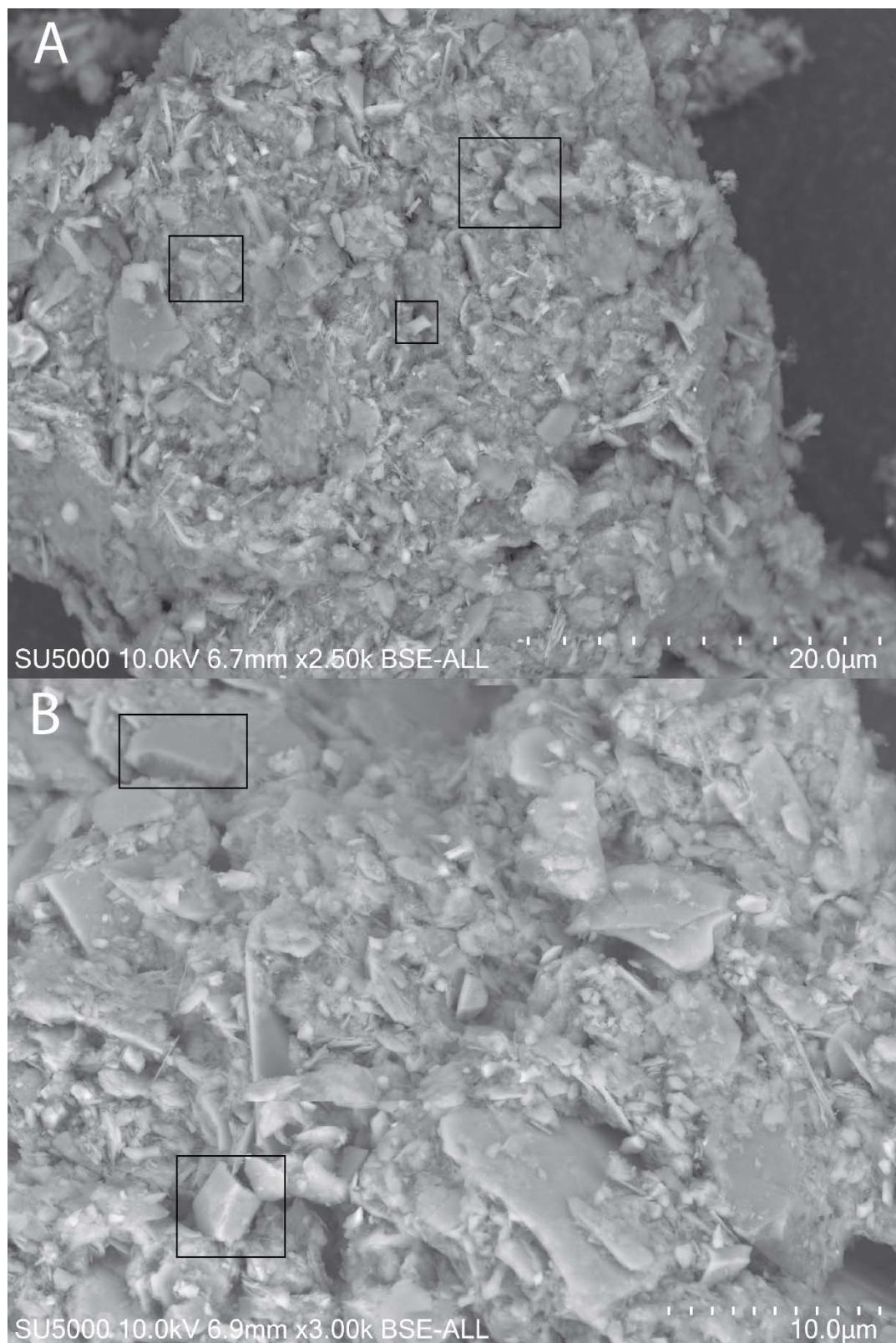


Figure 4 Possible secondary minerals in A. B60 and B. B80.

Appendix D

XRD & XRF ANALYSES



ANALYSEKONTRAKT NR.: 2020.0177
NGU PROSJEKT NR.: 021003

OPPDRAKGIVER: NGI

ADRESSE: Postboks 5687 Torgarden, 7485 Trondheim

TLF.: 94829497

KONTAKTPERSON: Priscilla Paniagua

PRØVETYPE: leire tilsatt brenkalk

ANTALL PRØVER: 4

IDENTIFIKASJON AV PRØVER: Ifølge liste fra oppdragsgiver

PRØVER MOTTATT DATO: 05.10.2020

TILSTANDEN TIL PRØVER VED MOTTAK: *ingen avvik*ANMERKNINGER: *Ingen***SPESIFIKASJON AV OPPDRAGET I HENHOLD TIL ANALYSEKONTRAKTEN**

BESTILTE ANALYSER	DOKUMENTASJON *)	OMFATTES AV AKKREDITERING
XRF-analyse av hovedelementer	LABdok_G01	Delvis
XRD: identifikasjon og kvantifisering av mineraler	LABdok_M01	Nei

Denne rapporten inneholder i alt 8 sider. For supplerende data som sendes i kun digitalt format se Anmerkninger.

Alle forhold ved prøvetaking, behandling og transport av prøvene før innlevering til laboratoriet er underlagt oppdragsgivers ansvar. Analyseresultater framlagt i denne rapporten refererer derfor kun til det prøvematerialet som er mottatt av laboratoriet.

Gjengivelse av analysedata skal skje på en slik måte at meningsinnholdet i rapporten ikke endres. Rapporten skal ikke reproduceres annet enn i sin helhet, uten godkjenning fra laboratoriet.

Trondheim, 29. oktober 2020

Analyserapport godkjent av¹:Ana Banica
Leder for laboratorier

*) For mer informasjon om metoder kontaktes laboratoriet.

INSTRUMENT: PANalytical Axios 4 kW XRF (Rh-røntgenrør)

METODE: XRF-analyse av hovedelementer. Metoden er beskrevet i LABdok_G01.

Analysene er utført på glasspiller fremstilt ved smelting av 0.6 g prøvemateriale blandet med 4.2 g litiumtetraborat ($\text{Li}_2\text{B}_4\text{O}_7$).

Prøvematerialet er forglødet ved 1000 °C før smelting, og analysedataene regnet tilbake til uglødet prøve.

MÅLEOMRÅDER, NEDRE BESTEMMELSESGRENSER (LLQ) OG ANALYSEUSIKKERHETER

	SiO_2^*	Al_2O_3^*	Fe_2O_3^*	TiO_2^*	MgO^*	CaO^*	Na_2O^*	K_2O^*	MnO^*	P_2O_5^*	Gl.tap*	Ba	Co	Cr	Cu	Ni	Pb	Sr	V	Zn	Zr	S	
LLQ:	0.5	0.04	0.04	0.01	0.1	0.03	0.1	0.02	0.01	0.01	0.05	50	30	50	20	20	50	10	30	30	30	200	
Enhet	Vekt %												mg/kg										
Måleområde 1:	0.5-5	0.04-0.5	0.04-0.5	0.01-0.1	0.1-1	0.03-2	0.1-1	0.02-0.5	0.01-0.1	0.01-0.2	0.05-1	50-300	30-50	50-100	20-50	20-50	50-200	10-50	30-50	30-100	30-100	til 15000	
Usikkerhet 1 (rel. %):	10	30	30	40	20	15	20	40	30	20	7	40	40	30	30	30	40	50	30	30	30	30	
Måleområde 2:	5-50	0.5-6	0.5-1	0.1-1	1-5	2-5	1-25	0.5-25	0.1-0.5	0.2-1	1-100	300-1000	50-100	100-25000	50-150	50-500	200-500	50-100	50-150	100-5000	100-500	kun semikvantitativ	
Usikkerhet 2 (rel. %):	5	10	10	10	10	10	5	5	10	10	3.5	20	20	15	15	10	20	15	20	10	20		
Måleområde 3:	50-100	6-25	1-55	1-10	5-50	5-100			0.5-5	1-10		1000-3000	100-200		150-2000	500-5000	500-5000	100-5000	150-1000		500-15000		
Usikkerhet 3 (rel. %):	2	5	5	5	5	5			5	5		10	10		5	5	10	5	10		10		

***) Akkreditert parameter. For eventuelle unntak se Anmerkninger**

De oppgitte usikkerhetene (\pm) representerer dekningsfaktor 2 (95 % konfidensintervall).

Sulfidbundet og elementært svovel vil avdampes under prøvepreparering. Verdier av svovel er derfor minimumsanslag.

Data for glødetap (Gl.tap) rapporteres uten å sette inn LLQ.

PRESISJON: Det analyseres rutinemessig kontrollprøver som føres i kontrolldiagram (X-diagram). Disse kan forevises om ønskelig.

Analysekontrakt nr.: 2020.0177

Prøvemateriale: GEOLOGISK MATERIALE

Antall prøver: 4

Anmerkninger: ingen

Delrapport med forsiden ("Forside_XRF_Hoved") og sider med analysedata ("XRF_Hoved"). Fullstendig analyserapport finnes kun i papirformat.

Gjengivelse av analysedata skal skje på en slik måte at meningsinnholdet i rapporten ikke endres.

Merk! Data i rapporten er skrivebeskyttet.

Prøver preparert av: Ann Elisabeth Karlsen

Rapportert av: Jasmin Schönenberger

Analyser fullført (dato): 28.10.2020

NGU-nr #	Prøve ID	SiO ₂ * [%]	Al ₂ O ₃ * [%]	Fe ₂ O ₃ * [%]	TiO ₂ * [%]	MgO* [%]	CaO* [%]	Na ₂ O* [%]	K ₂ O* [%]	MnO* [%]	P ₂ O ₅ * [%]	Glødetap* [%]	Sum Hoved [%]
	Stabila B40 + Standard med sement med 60 kg/m ³	45.2	17.0	9.86	0.748	6.26	5.20	1.68	3.93	0.111	0.123	8.12	98.2
	Stabila B60 + Standard med sement med 30 kg/m ³	45.9	17.2	9.92	0.760	6.32	4.04	1.73	3.95	0.112	0.117	9.30	99.3
	Stabila B80 + Standard med sement med 30 kg/m ³	45.5	17.3	10.1	0.763	6.42	4.12	1.69	4.07	0.113	0.118	8.86	99.1
	Stabila B100 + Standard med sement med 30 kg/m ³	45.6	16.6	9.55	0.735	6.03	4.10	1.69	3.81	0.108	0.121	11.3	99.7

NGU-nr #	Prøve ID	Ba [mg/kg]	Co [mg/kg]	Cr [mg/kg]	Cu [mg/kg]	Ni [mg/kg]	Pb [mg/kg]	Sr [mg/kg]	V [mg/kg]	Zn [mg/kg]	Zr [mg/kg]	S [mg/kg]
	Stabila B40 + Standard med sement med 60 kg/m ³	556	44	217	65	133	<50	179	163	163	116	585
	Stabila B60 + Standard med sement med 30 kg/m ³	601	30	208	77	132	<50	159	165	151	117	302
	Stabila B80 + Standard med sement med 30 kg/m ³	634	31	218	69	134	<50	158	176	156	114	459
	Stabila B100 + Standard med sement med 30 kg/m ³	522	38	202	61	124	<50	159	166	145	118	417



NORGES
GEOLOGISKE
UNDERSØKELSE

Leiv Eriksens vei 39
NO - 7040 Trondheim
Tlf.: 73 90 40 00
E-post: lab@ngu.no

XRD-analyse
GEOLOGISK MATERIALE
Analysekontrakt nr. 2020.0177

Instrument: BRUKER D8 Advance med Cu røntgenrør og Lynxeye XE detector.

Metoder: LABdok_M01: XRD-analyser

Analyseformål: Identifikasjon og kvantifisering av mineraler ved XRD

Oppdragsnummer: 2020.0177

Prosjekt nummer: eksternt oppdrag

Oppdragsgiver: NGI (Ana Priscillia Paniagua Lopez)

XRD scan: Cu K α , 40 kV/40 mA, scan 3-75°2 θ ; step size = 0.02 °2 θ ; time/step = 1 s; soller slits 2.5 °
fixed divergence slit 0.6 mm; Ni-filter; knife edge; rotasjon 1/30

Prøvetype: sediment

Antall prøver: 4

Nedre bestemmelsesgrense: Den nedre bestemmelsesgrensen er avhengig av mineral, men er vanligvis 1-2 vekt%.

Usikkerhet: Avhengig av prøvematerialet har Rietveld modellering en usikkerhet på minst 2-3 vekt%.

Kontrollrutiner: Kontrollprøve kjøres rutinemessig (2 θ -value/d-value) og resultatene registreres i kontrolldiagram (X-diagram).
Disse kan forevises om ønskelig.

Anmerkninger: Se kommentarer side 3.
Rådata av scan kan leveres på forespørsel.

Delrapport med forsiden ("Forside_XRD") og sider med analysedata ("Data_XRD") og tilleggsinformasjon ("Kommentarer_XRD"). Fullstendig analyserapport finnes kun i papirformat.
Gjengivelse av analysedata skal skje på en slik måte at meningsinnholdet i rapporten ikke endres.

Ferdig analysert	13.10.2020	Jasmin Schönenberger
	Date	Operatør/Datatolkning

prøve ID	qtz	plag	ill/musc	bt	chl	amph	cal	dol	rt	GOF	Rwp
Stabila B40 + Standard med sement med 60 kg/m³	12	18	23	22	14	6	4 *	1	spor	1.61	16.48
Stabila B60 + Standard med sement med 30 kg/m³	12	16	24	22	15	6	4	1	spor	1.61	16.6
Stabila B80 + Standard med sement med 30 kg/m³	12	16	24	22	15	6	4	1	spor	1.61	16.6
Stabila B100 + Standard med sement med 30 kg/m³	13	16	23	23	14	6	4	1	spor	1.54	16.22

* Det finnes litt mer (ca. 0.5 vekt%) kalsitt i prøve "Stabila B40 + Standard med sement med 60 kg/m³", selv om det ikke vises når man runder resultatene i Rietveld modellering.

Kommentarer om XRD analyser

Mineralidentifisering skjer med automatisk eller manuelt søk etter topp-posisjoner i BRUKER programmware Diffrac.EVA ver5.2. Både Crystallographic Open Database (COD) og PDF 4 Minerals fra ICDD (International Centre for Diffraction Data) brukes som databaser. Mineralkvantifisering gjøres med Rietveld modellering og programmware TOPAS 5.0. De kvantifiserte mineralkonsentrasjonene ble omberegnet til elementokside og verifisert mot XRF-data.

MERK! Prøvene ble ikke McCrone-møllet i isopropanol for å unngå reaksjon/hydratisering med tilsatt brentkalk. Prøvene var allerede veldig finkornet og ble møllet kun med hånd i agatmorter.

Det finnes litt mer (ca. 0.5 vekt%) kalsitt i prøve "Stabila B40 + Standard med sement med 60 kg/m³", selv om det ikke vises når man runder resultatene i Rietveld modellering.

Forkortelser:

GOF/Rwp	GOF betyr "goodness of fit" og angir sammen med <i>Rwp</i> pålitelighet av Rietveld modelering. [det gjelder omrent: GOF < 2.5: meget bra modelering; GOF < 3: pålitelig modelering]
Rwp	weighted profile factor

qtz	kvarts
plag	plagioklas
ill/ms	illitt/muskovitt
bt	biotitt
chl	kloritt-gruppe
amph	amfibol
cal	kalsitt
dol	dolomitt
rt	rutil

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