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Application of parallel plate rheology to investigate shear strength of sensitive clay treated with inorganic salts

Kamila Zabłocka, Klaartje De Weerdt
Department of Structural Engineering, Norwegian University of Science and Technology, Trondheim, Norway
kamila.zablocka@ntnu.no

Mohammad Hemayati, Ali Ghoreishian Amiri
Department of Civil and Environmental Engineering, Norwegian University of Science and Technology, Trondheim, Norway

Priscilla Paniagua, Sølve Hov
Norwegian Geotechnical Institute, Trondheim, Norway

ABSTRACT: The efficiency of the stabilising effect of KCl, KOH and K_2CO_3 on improvement of the mechanical properties of a remoulded Norwegian quick clay was investigated to gain insight into the stabilising mechanisms. The salts were added to the quick clay as salt solutions with anion concentrations that varied between 0.037 and 0.6 mol/L. The shear strength of the treated clays was determined by the falling cone (FC) test with a 10 g 60° cone and by parallel plate rheology (PPR), and the results were compared. The clay was defined to no longer be quick if the remoulded shear strength exceeded 0.33 kPa. At anion concentrations below 0.1 mol/L KCl treated clay achieved the highest strength increase out of the salts presented here, however, K_2CO_3 was found to be the most efficient binder at higher concentrations. The shear strength increase as a function of KOH concentration corresponded to that of K_2CO_3 indicating that similar improvement mechanisms are at work when these two salts are used to treat the clay. The results of the two testing methods were in agreement, however the rheological measurements were able to determine the shear strength with a higher level of precision compared to the FC tests. The former has a precision of 1E-6 kPa while the precision of the latter is 0.1 kPa. According to the FC results, all three salts improved the clay sufficiently such that it was no longer classified as quick clay. On the other hand, according to the rheological measurements, only a high concentration of K_2CO_3 resulted in a shear strength that significantly exceeded 0.33 kPa. KOH and KCl treated clays resulted in shear strengths around 0.3 kPa at similar anion concentrations.

KEYWORDS: quick clay, salinity, ground improvement, rheological properties, short term strength

1 INTRODUCTION

Marine quick clay is a remnant of the last ice age and can be found in the Nordic countries as well as northern parts of Canada, Alaska and Russia. Such clay happens to be prevalent in areas with high population density in Norway that are found below the marine limit. If construction is to take place it oftentimes must be preceded by ground improvement to prevent the rapid collapse and liquification of the quick clay once it is exposed to load. The mechanical properties of the clay can be improved by the addition of binders, traditionally, lime and cement (LC) (Larsson, 2021) however more environmentally friendly binders are increasingly being used as lime and cement has a large CO_2-footprint. Such clay is defined to be quick if the penetration depth in a falling cone tests with a 10 g 60° cone exceeds 9 mm in a remoulded sample, which correlates to remoulded shear strength (c_{ur}) that is equal or lower than 0.33 kPa according to ISO 17892-6:2017 (Norwegian Committee for Standardization, 1988; Swedish Committee for Standardization, 2017; Wiig, Strand and Haugen, 2020).

The present paper is part of a larger study on the efficiency of inorganic salts for the improvement of the mechanical properties of remoulded quick clay in the short-term (< 1 day) (Zabłocka et al., [In preparation]). The results can be used as a basis for recommendations for new environmentally friendly binders. Several studies have considered the effect of salt on the structure of quick clay, however the focus has been on the effect of NaCl on reference clay materials (Gustafsson et al., 2000; Ramos-Tejada et al., 2001; Tombácz and Szekeres, 2006; Jeong, Locat and Leroueil, 2012). Helle et al. (Helle, Nordal and Aagaard, 2018) looked at the effect of KCl on Norwegian marine quick clay however no comparison with other salts was conducted.

As the remoulded quick clay samples possess little to no loadbearing capacity, the falling cone (FC) test was chosen to determine the remoulded shear strength as opposed to the uniaxial compression test. The FC test was compared with the parallel plate rheological (PPR) measurement which has the potential to determine the shear strength in a precise and reliable manner. Although the uniaxial and triaxial tests are preferable testing methods when investigating new binders, they are not well-suited in this case as FC and PPR deal with residual strength only. Furthermore, both FC and PPR tests require less material than the uniaxial compression test, where several 100 g of material can be required (Norwegian Committee for Standardization, 2015). Various rheological measurements have been used previously to determine the mechanical properties of suspensions containing artificial reference clay materials in the presence of a stabiliser or binder (Gustafsson et al., 2000; Ramos-Tejada et al., 2001; Tombácz and Szekeres, 2006; Jeong, Locat and Leroueil, 2012). However, little is known about the rheological properties of natural clay materials in samples with a high concentration of solids can change the properties of the clay significantly (Khaldoun et al., 2009).

The comparison between the considered geotechnical laboratory testing methods and PPR is given in Table 1.

Table 1: Estimates for samples size, duration, range and precision of various methods used to measure shear strength. Note that the values are given for the FC test using a 10 g 60° cone as this is the recommended cone for quick clay for c_{ur} determination.

Method	Sample weight [g]	Duration [min]	Stress range [kPa]	Precision [kPa]
Falling cone	60	2	0.1 – 1.5	0.1
Triaxial	300	1440	>5	1E-4
Uniaxial	300	10	>5	0.1
Parallel plate rheology	10	2	1E-6 - 1	1E-6

The aim of this paper is two-fold. Firstly, the novel parallel plate rheological method was used to determine the improvement effects of various salts in quick clay samples with a high concentration of solids. Secondly, the results from the FC tests and the rheological measurements were compared to determine the suitability of the latter as a geotechnical method for mechanistic studies which require a high level of precision.

The effects of KCl, KOH and K_2CO_3 on the residual shear strength of quick clay were investigated using the FC test and parallel plate rheology. The salts were added to the quick clay in the form of salt solutions and their concentrations were expressed in mol/L to ensure an equal basis for comparison. The anion concentrations of the solutions varied between 0.037 and 0.6 mol/L, the latter is similar to the salt concentration in the Atlantic Ocean (Moon, 1972).

2 EXPERIMENTAL APPROACH

2.1 *Materials*

The clay studied in this paper was extracted from the Tiller-Flotten quick clay site which is a part of the Norwegian GeoTest site (NGTS) project (Kåsin et al., 2019) located approx. 10 km south of Trondheim, central Norway. The extraction depths were approx. 10 and 13 m. The clay at these depths has been classified as very sensitive quick clay (L'Heureux et al., 2019). The oxide and mineral compositions have been reported previously by Hov et al. (2022).

KCl, KOH and K_2CO_3 are the salts considered in the present study; however, a larger selection was investigated and will be presented elsewhere (Zabłocka et al., [In preparation]). The salt solutions were prepared by adding deionized water to analysis grade salts, i.e. salts with a purity of at least 99%. The balance used to control the quantity of deionized water was a Mettler Toledo's PM2000 Balance with a precision of 0.01 g. Ohaus's Pioneer PA413C Balance with precision of 0.001 g was used to measure out the correct quantities of the salts.

2.2 *Sample preparation*

Piston sampling was used for the clay at 13 m using Ø54 mm metal cylinders and block sampling was used to extract the clay at 10 m.

The clay batch was remoulded and homogenized to make it more representative and homogeneous before samples were prepared. For this purpose, the clay was mixed with a Kenwood's Major Titanium mixer with a K-paddle on speed setting "1" for 30 seconds. The clay was then placed in a sealed and airtight 5 L plastic container for storage at 20 °C.

The natural water content of the clay has been reported to be approx. 40% (L'Heureux et al., 2019). For the purposes of this study, the water content of the clay samples was adjusted to be approx. 50%. The water content was determined regularly according to the NS-EN ISO 17892-1:2014 (Norwegian Committee for Standardization, 2015) and adjusted based on weight using deionized water. After adjustment of the water content, the clay was mixed by hand using a spatula.

The clay was treated with a quantity of salt solution that corresponded to 25% of the total weight of the sample, e.g. 9 g of quick clay with a water content of approx. 50 % was treated with 3 g of salt solution. The salts were added to the clay in the form of solution to ensure correct salt concentration in the sample. The treated clay was mixed for 1.5 min using Stuart's SA6 Mini Vortex mixer immediately after weighing and left to cure in small, sealed plastic containers for approx. 20 hours before testing at 20 °C. The preparation was the same for the PPR and FC tests, however, the FC tests required 60 g of quick clay for each sample, while for the PPR tests only approx. 10 g quick clay per sample was needed.

2.3 *Methods*

2.3.1 *Rheological measurements*

The rheological measurements (PPR test) were conducted using an Anton Paar's Physica MCR 300 rheometer with serrated parallel plate geometry with a diameter of 50 mm, see Figure 1. Approx. 2.5 ml of the treated clay was placed on the stator and trimmed such that the 1 mm gap between the plates was filled after the rotor was lowered into measurement position. A moisture trap was placed around the sample to prevent it from drying out during the measurement. The rheometer imposed a constant temperature of 20 °C on the treated clay during the measurement. The resistance was calculated automatically by the software based on the geometry used in the tests, i.e. the parallel plates, and the detected rotation of the upper plate.

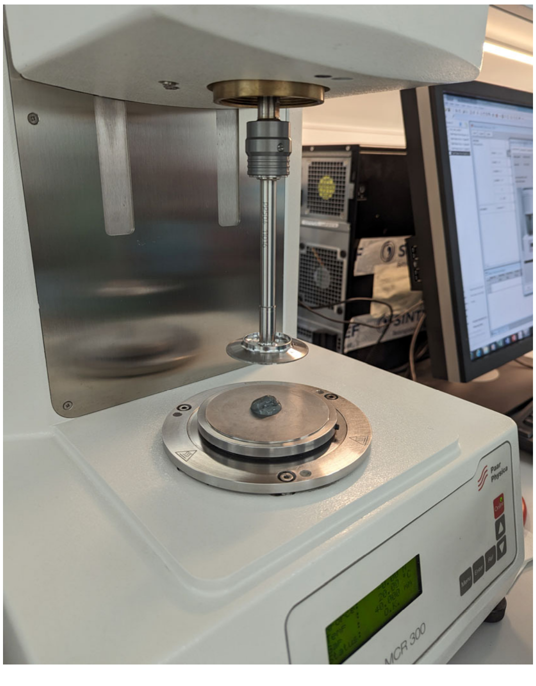

Figure 1: Anton Paar Physica MCR 300 rheometer with serrated parallel plate geometry.

The measurement sequence consisted of several intervals which can be characterized by an imposed effect, i.e. shear stress or shear rate. In this paper, one such interval, the upwards flow curve, will be discussed. The remaining intervals will be presented elsewhere (Zabłocka et al., [In preparation]). The upwards flow curve is characterized by a shear rate which increases linearly from 0 to 100 s^{-1}, i.e. 0 to 6 000 rpm, and is imposed by the controlled rotation of the upper plate. The shear rate increased incrementally in the steps of 0.5 s^{-1}, and the response was logged every 0.5 seconds. As a result, the measurement of the upwards flow curve took approx. 1.5 minutes. The first value of shear stress logged during this interval is considered the dynamic yield shear strength of the material. This parameter can be said to represent the shear strength of the static structure that the clay particles form.

2.3.2 *Falling cone (FC) test*

The FC test was conducted on a selection of the treated clays. A 10 g 60° cone was dropped six times on the surface of each treated clay sample, according to NS-EN ISO 17892-6:2017 and illustrated in Figure 3. The FC shear strength was calculated based on the ISO 17892-6:2017, Norwegian and Swedish standards (Norwegian Committee for Standardization, 2011; Swedish Committee for Standardization, 2017), the relation between the penetration depth and shear strength for the cone used in this study is given in Figure 2.

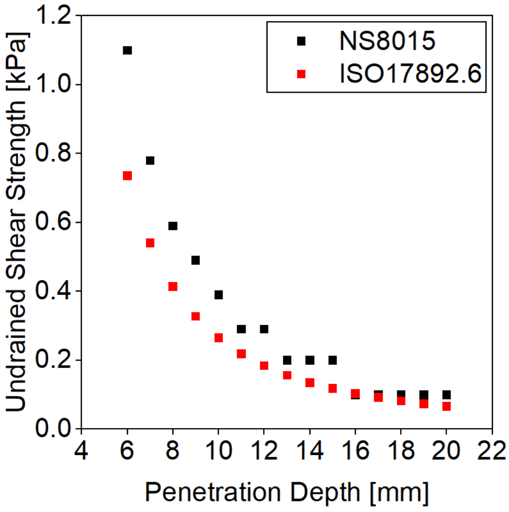

Figure 2: The determination of FC shear strength as a function of penetration depth using the 10 g 60° cone during the FC test according to the Norwegian and ISO standards (Norwegian Committee for Standardization, 1988; Swedish Committee for Standardization, 2017).

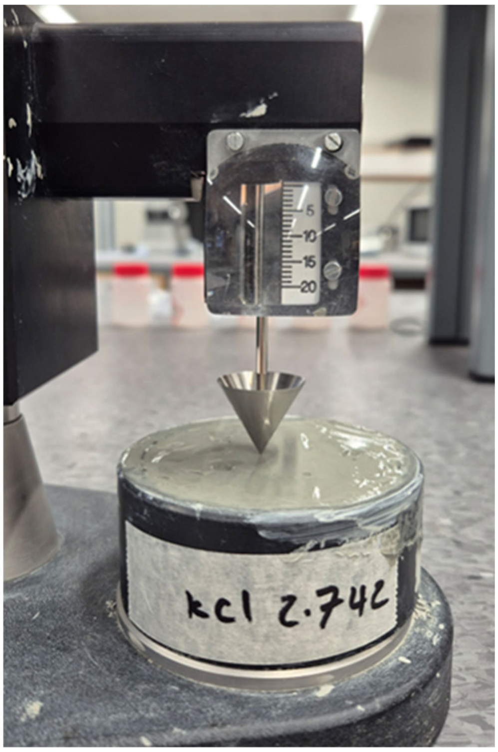

Figure 3: Picture of the falling cone apparatus and the 10 g 60° cone used during the FC tests. (Photo: Mohammad Hemayati)

3 RESULTS

Figure 4 shows the response of two treated clay samples during the upwards flow curve and illustrates how the dynamic yield shear strength was determined in the PPR test. Both samples exhibit clear non-Newtonian behaviour(Tsugawa et al., 2020), as the yield shear stress is not zero. In addition, the clay sample treated with a high concentration of KCl (red) shows a shear-thinning behaviour at high shear rates, as indicated by the curve levelling out as the shear rate increases. This implies a decrease in apparent viscosity, which is defined to be the slope of the curve (Tsugawa et al., 2020). The clay treated with low concentration of KCl (green) on the other hand shows plastic behaviour, i.e. the shear response is approx. constant with respect to shear rate.

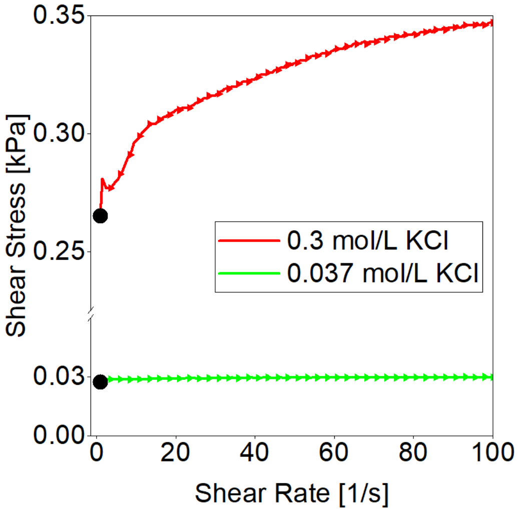

Figure 4: The rheological results from PPR tests of the upwards flow curve for clay samples treated with 0.3 and 0.037 mol/L KCl. The

black points indicate the dynamic yield shear strength. Note that the y-axis is split between 0.05 and 0.255 kPa.

The repeatability and time dependency of the PPR tests was investigated by measuring five samples treated with the same salt solutions, the results are given in Table 2. The results show an average dynamic yield shear strength of 56.7 Pa and a relative standard deviation (RSD) of 4.5%.

Table 2: The dynamic yield shear strength of clay samples treated with 0.3 mol/L NaOH after various curing times.

Approx. curing time [h]	Dynamic yield shear strength [Pa]
19	58.7
19.5	58.7
20.5	57.4
22	56.7
24.5	51.8

Figure 5 and Figure 6, respectively, show how the shear yield strength changes as the anion and cation concentrations of the added salt solutions increase. Each point corresponds to one clay sample treated according to the procedure described in Section 2.2. The clay sample with the anion concentration equal to 0 mol/L was prepared in the same manner, however distilled water was used in place of salt solution.

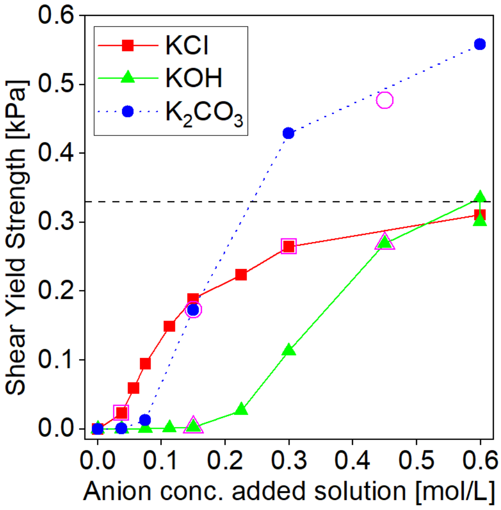

Figure 5: Change of shear yield strength from PPR tests as a function of anion concentrations of the added salt solutions. The solid and dotted lines indicate clay from 13 m and 10 m respectively. Pink symbols indicate treated clay samples chosen from FC tests. The black dashed line indicates the strength requirement for classifying the clay as non-quick according to NS-EN ISO 17892-6:2017 (Swedish Committee for Standardization, 2017).

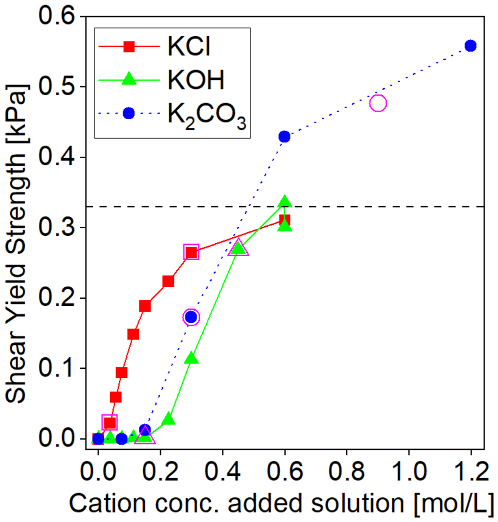

Figure 6: Change of shear yield strength as a function of cation concentrations of the added salt solutions. The solid and dotted lines indicate clay from 13 m and 10 m respectively. Pink symbols indicate treated clay samples chosen from FC tests. The black dashed line indicates the strength requirement for classifying the clay as non-quick according to NS-EN ISO 17892-6:2017 (Swedish Committee for Standardization, 2017).

Figure 7 show the estimates of the FC shear strength based on the FC tests according to the Norwegian (semi-transparent) and Swedish (opaque) standards. The pink symbols represent the dynamic yield shear strength determined from the PPR tests on of the treated clay samples. The salt concentration of the treated samples was chosen based on the result of the rheological measurements as indicated by pink symbols in Figure 5.

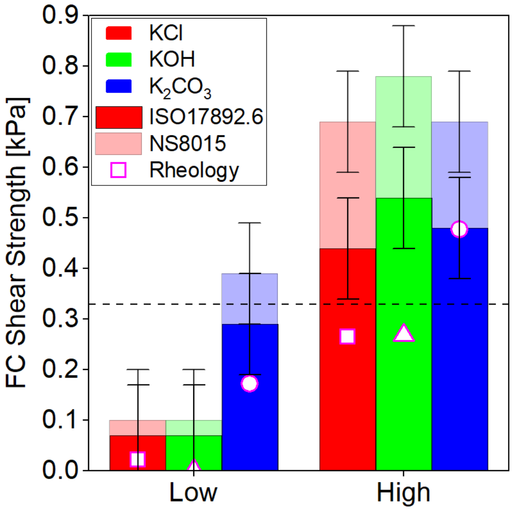

Figure 7: The FC shear strength determined by the FC test compared to the dynamic yield shear strength from PPR tests (pink symbols). The semi-transparent and opaque bars show FC shear strength calculated based on the NS8015 and ISO17892.6 standards, respectively (Norwegian Committee for Standardization, 2011; Swedish Committee for Standardization, 2017). The black dashed line indicates the strength requirement for classifying the clay as non-quick according to NS-EN ISO 17892-6:2017 (Swedish Committee for Standardization, 2017). “Low” and “High” indicate the relative salt concentrations from Figure 5.

4 DISCUSSION

4.1 *Improvement effect of the salts*

The increase in salt concentration leads to an increase in shear yield strength for all three salts considered, which can be seen in the results of the rheological measurements, see Figure 5 and Figure 6. This improvement is further verified by the FC test, see Figure 7. For concentrations below approx. 0.15 mol/L, the strength increase due to the addition of KCl is the largest out of the three salts. KCl has an improvement effect already at the lowest concentration considered, 0.037 mol/L, however at higher concentrations the increase in yield strength seems to reach a plateau. The strength increase between 0.3 mol/L and 0.6 mol/L is much smaller than that when KOH or K_2CO_3 is added in similar concentrations.

The effects of additions of KOH and K_2CO_3 appear similar when plotted against the cation concentrations as given in Figure 6, however there is a clear difference when anion concentration is considered, see Figure 5. The added solutions need to have a certain salt concentration before significant strength increase can take place, see Figure 5 and Figure 6. The anion concentrations are approx. 0.15 and 0.08 mol/L for KOH and K_2CO_3 respectively. These anion concentrations coincide with the same concentration of the cation (K^+) for both salts, approx. 0.15 mol/L, see Figure 6. The strength increases due to both of these salts overtake that of KCl, see Figure 5. It is noteworthy that both KOH and K_2CO_3 increase the pH of the treated sample, which KCl does not. The effect of the pH and the role of the stabilising effect of the cations will be investigated in a future study (Zabłocka et al., [In preparation]).

4.2 *Falling cone test*

As can be seen in Figure 7 there is a difference between the FC shear strength determined according to the NS8015 and ISO17892.6 standards. This is especially apparent for clay samples labelled “High” i.e. clay samples treated with solutions with a high salt concentration. This is a known and expected disparity due to the nature of the models described in Appendix A of the Norwegian standard (Norwegian Committee for Standardization, 1988). The Swedish standard gives a more conservative estimate of the FC shear strength while the Norwegian model gives a higher value due to an offset.

4.3 *Rheology as a geotechnical testing method*

The use of parallel plate rheology (PPR) is not widespread in the field of geotechnics; however, it does pose some advantages over more common testing techniques, especially when conducting fundamental research into the improvement mechanisms of quick clay.

The rheological measurements require much less material, 10 g, compared to traditional geotechnical tests, between 60 and several 100 g. This allows for a large experimental matrix while using one batch of clay, which reduces the introduction of unknow variables into the already complex system of raw quick clay. The tests were also conducted in a testing machine ensuring that the loading and logging of results was identical for all tests. Such measurements can be done by anyone who has access to a parallel plate rheometer and are not operator dependent, FC on the other hand requires experience to yield results of good quality as manual reading of the results on a mm scale is necessary. The measurement sequence was also quick, approx. 1.5 min, which made the experimental work efficient.

The rheological method makes it possible to investigate short-term strength (1 day or less) of treated clay which does not possess enough loadbearing capacity to stay intact in preparation for a standard uniaxial or triaxial test. This is also a limitation however, since samples with high shear yield strength or high stiffness cannot be measured using parallel plate rheology. This applies, for example, to clay treated with LC that has undergone curing for longer than a few hours. Reliable measurements of rheological properties can be achieved only on homogeneous and smooth samples that possess a level of workability. Such samples are also good candidates for the FC test however it is rarely conducted on stabilised samples, as they usually undergo sufficient curing to be tested in, more precise, standard compressive tests. This curing time however commonly exceeds 7 days which is outside the scope of this study.

The RSD of the dynamic yield shear strength determined by the upwards flow curve using parallel plate rheology was determined to be 4.5%, based on a series of five clay samples treated with the same salt solution, see Table 2. This is similar to the FC test where the average RSD of penetration depths was determined to be 3.8%. The precision of the rheological measurements, however, is superior to the FC test as a shear stress as low as 1E-6 kPa can be detected. The precision of the FC test depends on the operator, the instrument used and the consistency of the sample. The shear stress can only be determined with a precision of up to 0.1 kPa, see Figure 2, which is not sufficient for the fundamental research aimed at stabilisation mechanisms presented here.

5 FURTHER RESEARCH

To truly understand the stabilisation mechanisms of quick clay further research needs to be conducted into the effects of different anions and cations on the clay structure. Mechanisms such as cation adsorption, surface charges of the clay particles and the effect of pH will be investigated in (Zabłocka et al., [In preparation]).

A more comprehensive series of the FC tests will also be completed to further investigate a correlation between the FC tests and the rheological measurements.

6 CONCLUSION

6.1 *Effect of the salts*

The efficiency of the stabilisation mechanisms of KCl, KOH and K_2CO_3 were investigated using falling cone tests and parallel plate rheological measurements. The comparison was done based on residual shear strength determined by the two tests.

KCl was the most effective stabiliser at anion concentrations below 0.1 mol/L, however the rate of strength increase seemed to strongly diminish with concentration above 0.3 mol/L. KOH and K_2CO_3 needed to reach a higher concentration before their positive impact on yield shear strength became evident. They did not appear to lead to a plateau in shear yield strength in the concentration interval considered.

Only K_2CO_3 was able to result in a shear strength, approx. 0.5 kPa, that significantly exceeded the strength criterion for “non-quick” clay, 0.3 kPa, according to the rheological measurements. The other salts reached a maximum shear strength of around 0.3 kPa at highest salt concentrations.

All salts succeeded in stabilising the quick clay according to the falling cone test with similar results in terms of strength, between 0.6 and 0.8 kPa. According to parallel plate rheology only the sample treated with K_2CO_3 should have been classified as non-quick while the residual shear strength of the others was measured to be approx. 0.25 kPa, i.e. just below the strength criterion for non-quick clay.

6.2 *Parallel plate rheology as a geotechnical testing method*

The aim of this paper was to introduce parallel plate rheology as an alternative method of determining the short-term shear strength of quick clay stabilised with inorganic salts. The rheological measurement was compared to the falling cone test with the following conclusions:

- The rheological measurements require significantly less material (10 g) than traditional geotechnical tests (between 60 g and several 100 g) allowing for a large experimental matrix which is advantageous when investigating rheological parameters on one batch of quick clay.
- The duration of the rheological measurement and the falling cone test is similar, i.e. a few minutes.
- The rheological measurements gave reliable results with a degree of reproducibility that is similar to the falling cone test, RSD of 4.5% and 3.8% respectively.
- The shear strength can be determined more precisely using parallel plate rheology compared to the falling cone test, precision of 1E-6 kPa and 0.1 kPa, respectively. This is especially evident for samples with low shear yield strength.
- Only samples with low shear yield strength should be tested using parallel plate rheology which is a limitation of the method.

7 ACKNOWLEDGEMENTS

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