Appendix 3.4: Laboratory methods

Moisture content

Moisture content for the field samples was determined following the New Zealand Standard Methods of Testing Soils for Engineering Purposes (NZS 4402: 1986). Samples were dried at 105 °C and the moisture content was then determined by **Equation 1**:

$$Moisture\ content$$
$$= \frac{Mass\ of\ container\ and\ wet\ soil - mass\ of\ container\ and\ dried\ soil}{Mass\ of\ container\ and\ dried\ soil - mass\ of\ container}$$
$$\times 100 (\%)$$

Equation 1

Bulk density

Bulk density was determined following the linear measurements procedure outlined in ISO 17892-2 (2004). Four cylindrical tubes of known volume were carefully hammered into the soil face. The soil was trimmed off the ends of the tube and the bulk density was then defined by **Equation 2**:

$$\rho = \frac{mass\ of\ container\ and\ core - mass\ of\ sampling\ tube}{volume\ of\ tube}\ (kgm^{-3})$$

Equation 2

Dry bulk density could further be calculated using soil water content results by Equation 3:

$$p_d = \frac{100\rho}{100 + water\ content\ of\ the\ soil}\ kgm^{-3}$$

Equation 3

Particle density

The density bottle method employed by Head (1992) was chosen over other density bottle methods (e.g. Vickers, 1978; NZS 4402, 1986) as it was the most recently published and practicable due to the small grained (< 2 mm) particle sizes of the soil in question. Samples were disaggregated by hand rather than using a riffle box as Head (1992) denotes in step 2. Following drying and de-airing of the soil, the soil was crushed by hand so that it could be funnelled into the 50 ml pycnometers. The method then involved determining the particle density from the weight of the weighing bottle, the de-aired distilled water and bottle weight and finally the de-aired distilled water, soil and the bottle using **Equation 4**.

$$\rho_s = \frac{soil, bottle\ and\ stopper\ weight - bottle\ and\ stopper\ weight}{(bottle,\ stopper\ and\ deaired\ water - bottle\ and\ stopper\ weight) \atop -(bottle\ stopper\ and\ deaired\ water - soil, bottle\ and\ stopper\ weight)}\ (kgm^{-3})$$

Equation 4

Porosity

Porosity (%) was determined by dividing the dry bulk density by the average particle density, and is expressed as a percentage (**Equation 5**) (McLaren & Cameron, 1996).

$$Porosity\ (n) = \ 1 - \frac{dry\ bulk\ density\ (kgm^{-3})}{particle\ density} \times 100\ (\%)$$

Equation 5

Voids ratio

Voids ratio (no unit) is given by the inverse of porosity (Lancellotta, 1995) by **Equation 6**:

$$e = \frac{V_v}{V} = \frac{n}{1-n}$$

Equation 6

Atterberg limits

Atterberg Limits are widely documented standards, used to test the water content at which soils change state. They also are general indicators of what clay minerals may be dominating behaviour in the soil. Prior to testing, the soil was remoulded with steel spatulas on a glass plate until a consistent paste was achieved.

Liquid limit

The liquid limit is the water content at which a soil passes from a plastic to liquid state (Head, 1992; ISO17892-12, 2004). The now widely adopted fall-cone penetrometer test was chosen over the Casagrande method. This is because (1) the fall cone penetrometer test is more reproducible, and (2) it produces a narrower range of liquid limit values (Wires, 1984; Selby, 1993). For this test, the liquid limit is defined as the moisture content at which a 30°, 80 g drop cone penetrates 20 mm into the remoulded soil (Sherwood & Ryley, 1970; ISO 17982-12, 2004). Following remoulding of the soil with spatulas, a small sample was dried to determine initial moisture content. The first cone penetration test was carried out on the remoulded soil paste. A small amount of water was thoroughly mixed into the soil, and another small sample was taken to determine moisture content of this. Cone penetration and moisture

content was carried out on progressively wetter soil, until four sets of results at different moisture contents was obtained. These were plotted on a graph of penetration against moisture content.

Plastic limit

The plastic limit is the moisture content at which the soil transitions from a plastic behaviour to solid behaviour. The plastic limit was determined by the procedure outlined in NZS 4402 (1986). The method involves rolling 3 mm threads of remoulded soil paste on a flat surface, using evenly distributed pressure between the tips of the fingers and the first joint. The point at which the 3 mm threads initially shear longitudinally and laterally during rolling is considered to be the plastic limit. The threads were then immediately weighed and oven dried at 105 ° for 48 hours to determine the plastic limit.

Plasticity index

The plasticity index is the range of water contents between the liquid and plastic limits (Head, 1992; Selby 1993). The plasticity index was calculated using Test 2.4 of NZS 4402 (1986) (**Equation 7**).

$$Plastcity\ index\ (PI) = liquid\ limit\ (LL) - plastic\ limit\ (PL)(\%)$$

Equation 7

Activity

Activity is the ratio of the plasticity index to the percentage of clay sized particles. This is a measure of the plasticity of the clay sized particles. An estimation of the dominant clay mineral species can be estimated by the activity (**Equation 8**):

$$Activity = \frac{PI}{\%soil\ < 0.002\ mm}$$

Equation 8

Liquidity index

The complete remoulding of the soil destroys all *in situ* fabric therefore natural moisture content is lost. The liquidity index (**Equation 9**) is a ratio which is used to compare the soils plasticity with its natural field-moisture content (Selby, 1993). If the liquidity index (LI) = 100%, the natural soil is at its liquid limit, and if LI = 0 the soil is at its plastic limit (Selby, 1993).

$$Liquidity\ index = \frac{Natual\ moisture\ content}{PI}$$

Equation 9

Particle size

The University of Waikato Standard Operating Procedure method was used to find particle size. This method is based on Konert & Vandenberghe (1997). The method involves a series of steps, which are briefly outlined below.

1. **Drying and sieving**

   Drying and sieving was undertaken if soil was found to contain grains >2 mm.

2. **Organic matter digestion**

   The second stage involves pre-treatment of the sample to remove any non-silicate components including organic matter, carbonate and Fe-oxides (Keiser *et al.* 2014). Five representative soil samples of approximately half a teaspoon size were taken from the bulk sample and put into five pre-labelled 50 ml beakers. Beakers were placed in a fumehood cupboard before adding sufficient 10% hydrogen peroxide to cover the sample. Hydrogen peroxide gradually digests organic matter in the samples which can be observed as a fizzing of tiny bubbles. Significant amounts of iron oxide prolong the digestion period, making it difficult to determine the cut-off time for true organic matter digestion. 2 weeks was considered sufficient time for digestion. 5 ml of hydrogen peroxide was further added before the sample was heated gently on a hotplate. 1-2 drops of amyl alcohol was added to reduce frothing. The sample was boiled down to approximately 5 ml of slurry.

3. **Chemical dispersal:**

   10 ml of 10% calgon was added before leaving to cool overnight.

4. **Physical dispersal:**

   Samples were then sonicated in an ultrasonic bath for 5 minutes in order to disaggregate the sample and prevent mixing so that a representative sample could be taken for particle size analysis with the Malvern Mastersizer.

   There are two theoretical models which can then be applied to the results; the Fraunhofer theory and the Mie theory. The Malvern Mastersizer operates under the Mie theory. The Mie theory combines the known particle indices of both refraction and absorption, and based on these, estimates the particle sizes based on the light scattering behaviour when they are filtered through the suspended

medium (water). Sperazza *et al.* (2004) suggests the Mie theory is optimal as it less susceptible to grain size miscalculations.

Calculating the best absorption indices, including the refractive index (RI) and absorption (A) for the sample is crucial in estimating the particle size (Wyatt, 2009). When adjusting the RI from 1.37 to 1.56, the percentage of particles < 2 μ (clay sized) increased from 4.81 % to 80.75 %. Wyatt concluded that an A value of 0.01 and RI between 1.52 and 1.57 to be representative of soil. Following Wyatt's testing, a specified SOP or standard operating procedure for soil particle size was defined, where A was 0.01 and RI was 1.52. These values were replicated in this thesis to reduce operator variability and also to allow better reproducibility between results.

Soil solution was added dropwise to the water medium. The obscuration of the laser for the particle sizer is based on the amount sample added to the water. To prevent re-scattering of light, obscuration of between 10 and 20%, which are the pre-set values on the machine, were achieved when adding the sample. Sperazza *et al.* (2004) advocates that light re-scattering occurs at greater than 30% obscuration.

Because the Malvern Mastersizer measures samples as wet dispersions in a water medium, there is potential for the soil solution to flocculate. A user-bias is introduced if the sample contains a wide range of particle sizes. This bias can be reduced by increasing the number of replicates.