

CHAPTER 4

EXPERIMENT

The most important elements to consider in designing an experiment is the method of measuring electrical resistivity of different types of samples, especially clay-rich sediment and can the experimental results be validated with data obtained in the field. To correlate laboratory and field data, the sediment and leachate samples used in the laboratory must be a representative sediment and leachate found in the field. Furthermore, the method of packing and soaking the samples inside the resistivity box must accurately stimulate the conditions in the field. In fact, all the experimental parameters must closely approximate the equivalent parameters in the field. Such correlation is not simple or straightforward, partly because electrical resistivity relies on so many parameters. All the samples must be studied very carefully and the parameters of each sample carefully matched to the conditions in the field.

This chapter explains how representative samples were chosen, how these samples were collected, how the physical and chemical properties were measured, and how the experiment was set up in the laboratory.

4.1 Collection and characterization of sediment samples

4.1.1 Sediment collection

To collect a representative sediment sample, a systematic sample method was set up in an attempt to classify the soil units and to estimate their degree of homogeneity.

Previous sampling in this area included investigation in the Mae-Hia area by the Thai-German Research project (WADIS) in cooperation with the Department of Mineral Resources and Chiang Mai University. This study were sampled in both shallow wells and monitoring wells that were drilled to establish a geological and

hydrodynamic model for the Mae-Hia disposal area. During the drilling, the stratigraphy of the well was recorded. However, the textural descriptions done in the field lacked sufficient detail for use in this thesis experiment. For this reason additional sediment sampling and description of the Mae-Hia area was needed before the sediment samples could be used in the experiment.

Although hand augured borings require hard manual labor and are difficult at sites with heavy, compacted soils, they were nonetheless chosen because they are much less expensive than power-driven borings (Figure 4.1).

Even with additional sampling it is possible that the number and diversity of the samples may not be completely representative of the diversity of soil and sediment types in the field. Therefore, sampling error arises from the fact that the collected samples might include only selected sediment types.

Because of a lack drilling equipment and time, only twenty shallow auger drilling holes of 2 m depth were drilled. These sites were located to supplement the sampling data collected by the WADIS project. The locations of hand auger borings are shown in Figure 4.2, including the location of existing borings done by the WADIS project. Field data of each hand augured hole are included in Appendices B and C.



Figure 4.1 Sediment samples from a typical hand augur boring.

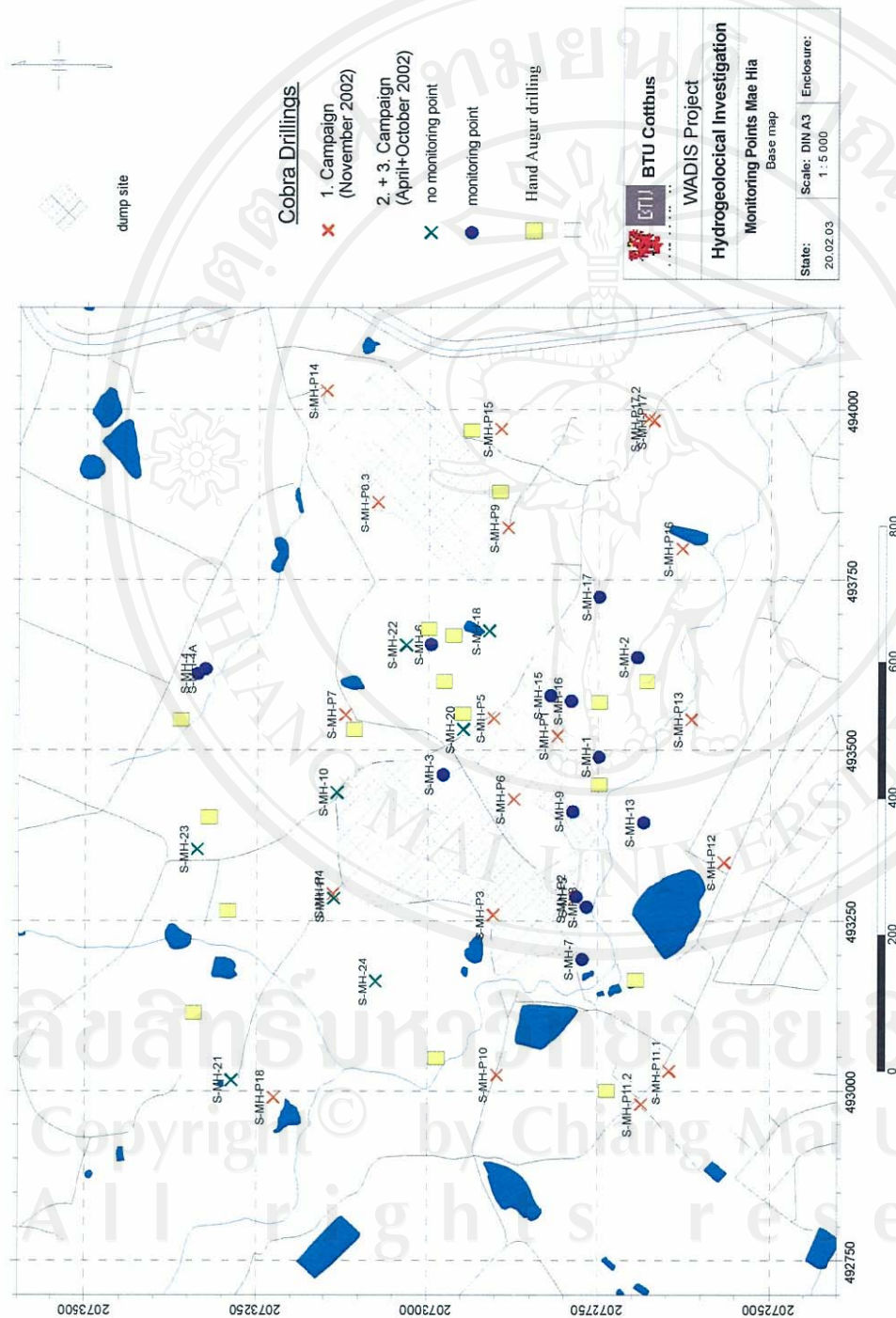


Figure 4.2 Map showing the locations of shallow hand augured borings made in this study, and the existing shallow cobra drillings made by the WADIS project (modified from Grisseman *et al.*, 2004).

Careful grain size analysis was done in the laboratory on samples from eleven holes. The samples contain with both coarse and fine grains in large amounts, so the test procedure used in this case is a combined analysis. After added distilled water to the sample, worked it into slurry and then washed the slurry through a #200 sieve. The coarse fraction caught by the sieve was oven dried, and the grain sizes determined by standard sieve analysis. The finer fraction that passed through the sieve was processed with both hydrometer analysis and the Shimadzu centrifugal particle size analyzer (Figure 4.3). This methods of grain size analysis followed those used by A.S.T.M. (1970) and outlined in the of Shimadzu centrifugal particle size analyzer (model SA-CP2-10) instruction manual (Shimadzu Corporation Kyoto, 1975).

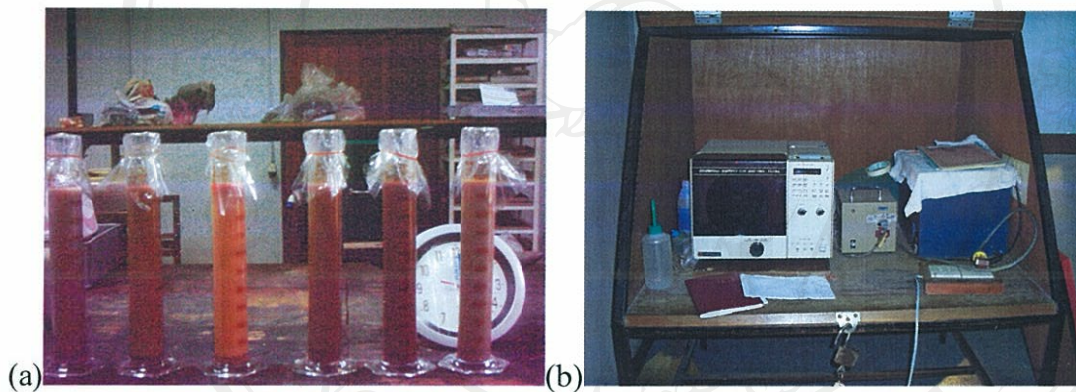


Figure 4.3 (a) Particle suspensions used in hydrometer analyses (b) The Shimadzu centrifugal particle size analyzer model SA-CP2-10.

Grain-size data of selected samples from the eleven borings made in this study are shown in Table 4.1. These sediments were classified based on ratios of the various proportions of the grain-size fractions. Definitions of the fractions conform to the standardized scale described in Wentworth grade scale (Figure 4.4).

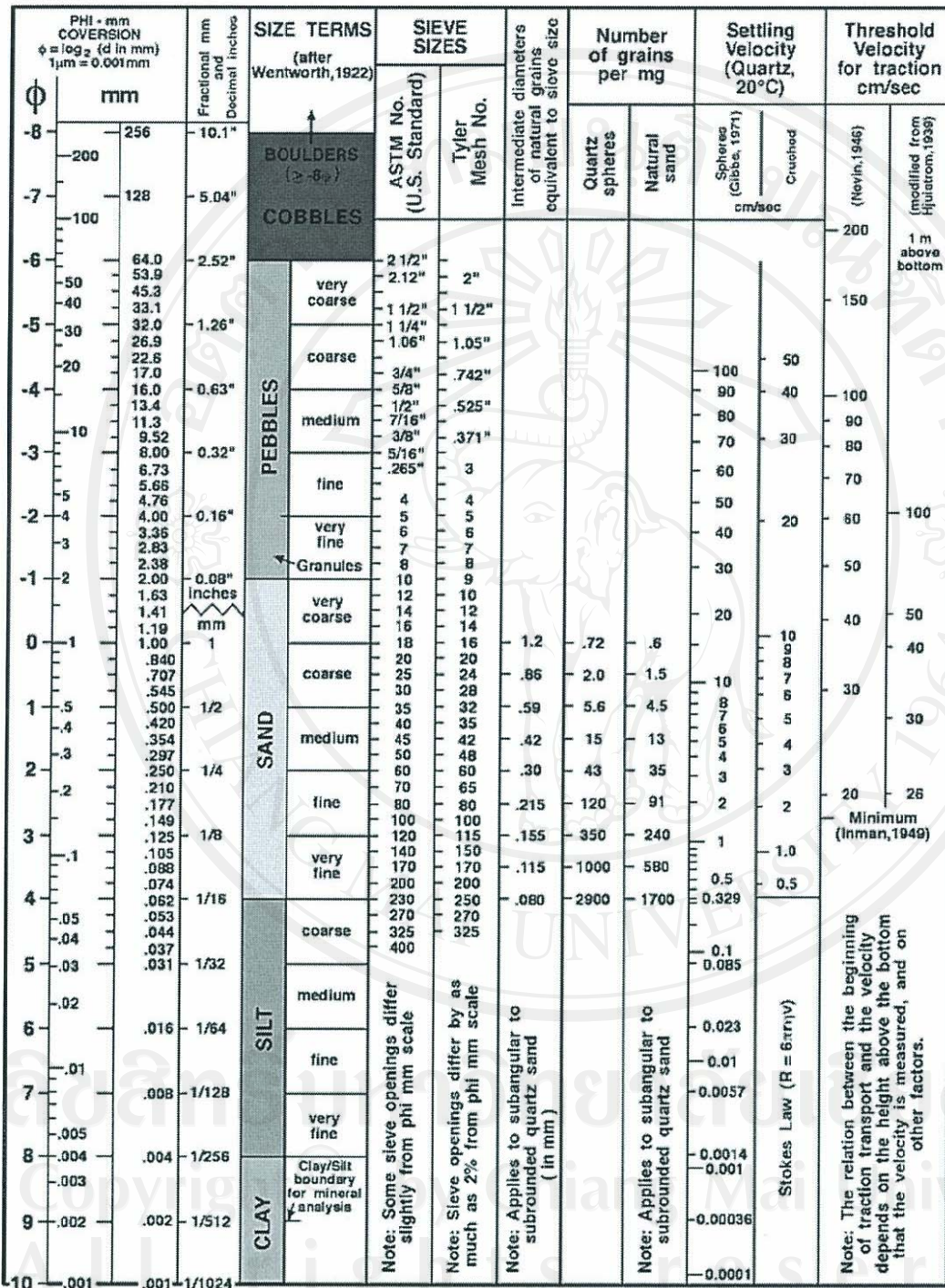


Figure 4.4 The Wentworth grade scale (U.S. Geological Survey, 2003)

Table 4.1 The grain size data from selected hand augured borings.

Sample No.	Depth (cm)	Grain size data (%)								
		pebble	granule	coarse sand	medium sand	fine sand	very fine sand	coarse silt	medium fine silt	clay
SMH-20	50-90	4.25	12.43	12.58	5.08	6.55	3.95	3.38	11.30	40.49
SMH-6 up	33-80	0	1.04	11.73	10.77	18.43	22.15	7.69	17.09	11.11
SMH-6 low	100-134	3.59	6.32	19.22	7.99	9.33	8.59	3.47	15.57	24.02
SMH-23 up	20-57	5.38	9.73	17.82	11.03	26.22	24.94	9.37	16.55	7.50
SMH-23 low	57-70	0.69	10.8	20.97	7.67	11.47	14.41	8.64	16.15	9.20
ST.18/2 up	42-75	0	1.12	11.60	10.37	18.21	19.1	12.49	17.21	9.90
ST.18/2 low	90-168	0.08	10.00	20.20	6.59	6.87	3.89	4.96	27.44	20.40
ST.18/3	58-105	0.29	4.89	19.43	12.25	10.31	9.59	7.20	22.89	13.13
ST.18/6	80-130	0.122	7.7	14.58	8.04	9.11	4.89	2.82	18.80	34.30
ST.18/7 up	85-130	1.06	5.90	8.08	5.67	7.09	10.69	3.84	34.22	23.44
ST.18/7 low	170-190	0	0.02	4.70	7.84	33.84	19.87	5.99	14.99	12.74
ST.18/8	0-50	0	4.75	32.42	19.52	8.36	4.59	3.04	14.67	12.64

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The sediment samples taken at various depths from eleven auger borings were considered as the suspect representative sediments of the area according to the existing data of WADIS project. Complied with their grain size analysis data, and then classified the samples by their ratios of sand to clay. The sample with the highest percentage of sand grain was considered as sand sample when the highest percentage of clay was considered as clay sample. Simple names were given to the types by considering the classifications done in the field by The WADIS project. So later on, the laboratory result will be practical correlated to the field data. Though there is a discrepancy in the nomenclature of those representative samples, as the cross plot of particle size distribution might not fall in the right texture categories in triangle diagram. By all those reasons, four samples of sediment were chosen as being representative of this study area.

- 1) Representative clay sample is from station 18/7 at the depth of 85-135 cm.
- 2) Representative sand sample is from station 18/8 at the depth of 50-100 cm.
- 3) Representative clayey sand sample is from station SMH-6 at the depth of 33-88 cm.
- 4) Representative sandy clay sample is from station 18/6 at the depth of 130 cm.

As four representative kinds of sediment; sandy, clayey sandy, sandy clay and clay, are commonly distributed around Mae- Hia landfill. From the systematic pilot auger drilling described above, the depth and locations of appropriately representative sediments can be found. At these locations, pits were excavated (Figure 4.5) and both disturb and undisturbed sediment samples were collected. The undisturbed samples were taken by using an aluminum core pushed into the ground (Figure 4.6). The samples were preserved carefully and labeled, and brought back to the laboratory.



Figure 4.5 (a) Top view of an opened trench (b) close up showing sediments exposed in the pit.



Figure 4.6 The push-core uses for collecting undisturbed samples.

4.1.2 Physical characteristics of sediments

Only representative samples from the field, were used for detailed physical and chemical analysis, the remaining samples were used later for further tests to investigate the relationship between electrical resistivity and the physical and chemical properties of contaminated material.

The physical properties that were analyzed were grain size, particle density, bulk density, porosity, and water content. The water content was measured as a volume fraction ranging from zero at after oven drying to a value equal to the porosity, when all pore spaces are saturated with water. Additionally, there are two intermediate states that occur as wet soil is dried. After a thoroughly saturated soil has drained by gravity for about two days, the water content at this stage is the “field capacity”. At a still drier state, the permanent wilting point, the water content is defined as the water content when the soil-water potential is -15 bars.

Field capacity and permanent wilting point are used for marking the upper and lower levels of the water content of the soil. Soil resistivity varied greatly below and above the permanent wilting point. At the permanent wilting point the water within the pore spaces is no longer continuous; as soon as a continuous film of water becomes interconnected through the pore spaces, the resistivity of the soil will increasing rapidly. For each sample, these two states of water content must be

determined before performing any experiments such that the variation of the saturation index in each sample can be considered during the experiment.

The particle size characteristics of four kinds of soils (Sandy, Clayey Sand, Sandy clay and Clay) were derived during reanalysis. Even though these locations were previously analyzed, but the collected sediment sample was much larger in volume; hence the grain size distribution may have changed. The grain size data and classification using Folk's classification system (Figure 4.7) are shown in Table 4.2. Soil size distribution curves for the four types of soils are shown in Figure 4.8. The remaining physical properties are summarized in Table 4.3

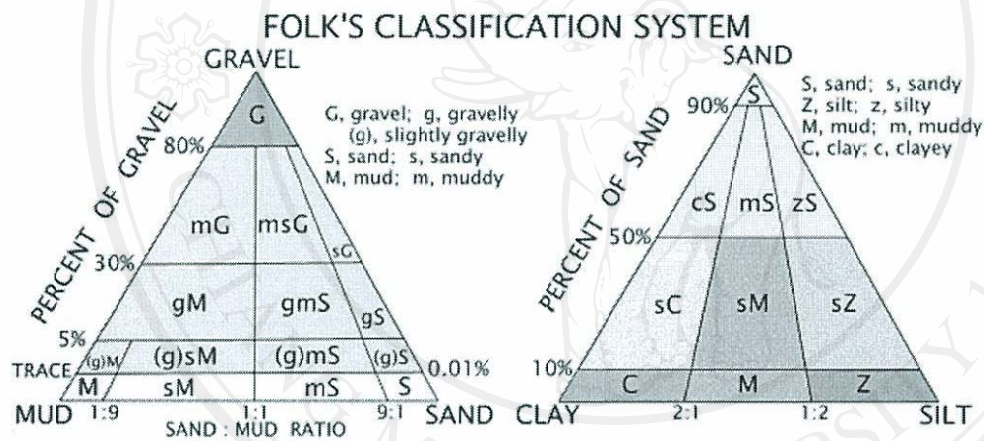


Figure 4.7 Folk's Classification system (U.S. Geological Survey, 2003).

Using the grain size distribution data, the classification the four types of soil was done on the Folk classification scheme. Note, however, that the sandy clay and clay fell into the same category in Folk's classification system. These two types of soil are very different, both in texture and color, and resulted in a different range of resistivity values. Perhaps variations in the type of clay minerals in the soil might be able to explain this wide resistivity variation. Doing clay mineral analysis is suggested in a future study.

Table 4.2 Grain size characteristics of sediment samples.

Sediment sample and Texture	Classification	Grain size distribution (%)				
		pebble	granule	sand	silt	clay
St.18/8 Sand	Gravelly Sand (gS)	1.63	5.60	90.99	1.01	0.57
SMH-6 Clayey sand	Muddy Sand (mS)	0.12	0.94	54.79	30.68	13.47
St.18/6 Sandy clay	Slightly gravelly Sandy Mud (g)sM	1.23	2.70	37.15	35.22	23.68
St.18/7 Clay	Slightly gravelly Sandy Mud (g)sM	0.43	2.58	29.63	46.83	20.55

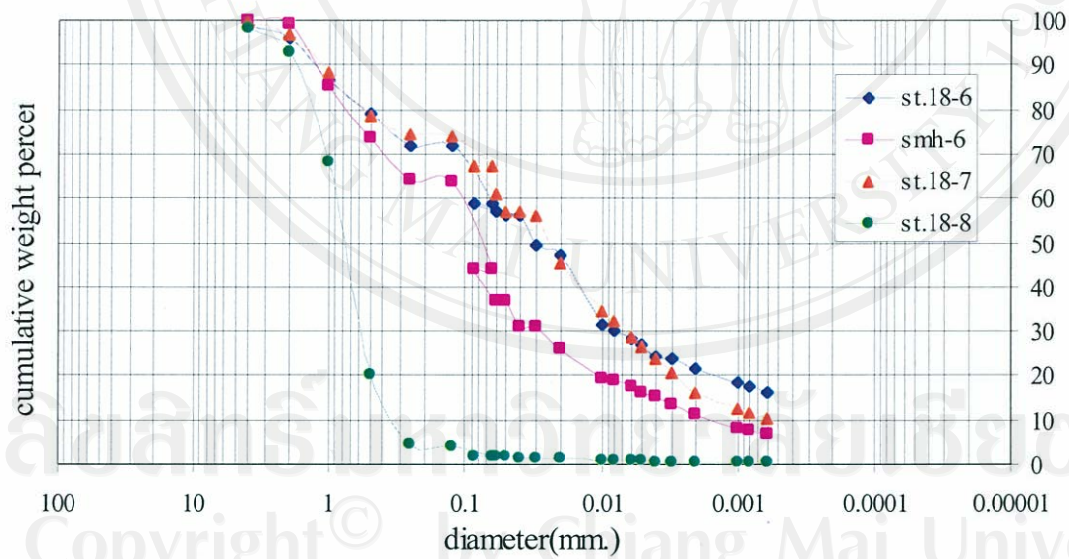


Figure 4.8 Soil size distribution curves for the four types of soils identified in this study.

Table 4.3 Physical properties of sediments.

Soil properties	St.18/8 (Sand)	SMH-6 (Clayey sand)	St.18/6 (Sandy clay)	St.18/7 (Clay)
Specific gravity (g/cm ³)	2.55	2.52	2.42	2.46
Bulk density (g/cm ³)	1.30	1.61	1.595	1.46
Porosity (%)	49.02	36.11	34.09	40.65
PWP (%)	0.997	2.88	9.03	7.87
FC (%)	2.51	10.26	20.57	21.20
Moisture content (%)	5.06	8.90	21.32	19.45
Saturation (%)	37.68	22.42	21.3	27.90

As can be seen in Table 4.3, the moisture content of all samples was higher than their field capacity. This means that the pore water has not completely drained downward to the water table. This is true even though all samples were collected at least 2-3 days after the last rainfall. It should be noted that even when samples are collected during dry weather, the water content of the sample may still exceed its field capacity level. In order to relate laboratory data to the field data, the saturation index of the samples must be considered very carefully.

4.1.3 Chemical properties of the sediments

Because the study area was used as a waste dump for more than 30 years, the sediment in the area is suspected to be contaminated by leachate. The contaminated sediment might contain high amounts of heavy metals and some of these can affect the electrical properties of the sediment. Therefore, the soils were analyzed for the major heavy metals before making any resistivity measurement in the laboratory. The chemical analysis of these soils was done using a Phillips 1410 X-ray Fluorescence Analyzer.

Table 4.4 Chemical composition (by % weight) of the sediment.

Chemical composition	Oxides	St.18/8 (Sand)	SMH-6 (Clayey sand)	St.18/6 (Sandy clay)	St.18/7 (Clay)
O	O	0	0	0	0
Na	Na ₂ O	2.686	1.429	0.943	1.679
Mg	MgO	0.289	0.371	0.485	0.867
Al	Al ₂ O ₂	11.283	20.707	34.951	24.096
Si	SiO ₂	79.246	69.988	54.649	65.672
S	SO ₂	2.178	2.411	2.094	1.444
K	K ₂ O	3.329	2.394	1.485	3.088
Ca	CaO	0.099	0.105	0.307	0.19
Ti	TiO ₂	0.097	0.433	0.564	0.438
Fe	Fe ₂ O ₃	0.773	2.09	4.487	2.407
Rb	Rb ₂ O	0.02	0.017	-	0.026
Zr	ZrO ₂	-	0.054	0.035	0.018
Mn	MnO	-	-	-	0.076

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4.1.4 Background electrical characterization of the sediment samples

During the resistivity measurements, the sediment sample must be soaked in the study fluid for a very long time period to make sure that the fluid has completely saturated the sample. During this period of time, it is possible that some chemical activity might cause a change in the electrical properties of the sample.

To test for any chemical changes during the soaking period, a small experiment was designed to test if the sediment had any significant reaction reacting with pore fluid while soaking. The specimens of 200 g, oven-dried samples and 200 ml of normal water were mixed. The specimens stayed seal to prevent evaporation or aeration at the laboratory temperature, before they were measured the electrical conductivity at intervals of one hour, 25 hours, 50 hours and 73 hours. Results are summarized in Table 4.5.

Table 4.5 Electrical conductivity ($\mu\text{S}/\text{cm}$) of the sample mixture.

Sediment sample	1 hr.	25 hrs.	50 hrs.	73 hrs.	504 weeks
St. 18/6	286	250	375	235	244
St. 18/7	114	132	128	120	141
St. 18/8	84	90	80	80	87
SMH-6	202	220	207	223	202

These results show that the conductivity of these samples stayed relatively constant during the total time of the experiment. Additionally, the conductivity of these samples correlates with the amount of metal oxide content of each sediment. For example, sample St. 18/6 had the highest Al and Fe-oxide content (as measured by XRF), and also had a larger conductivity than the other samples.

4.2 Leachate sampling and characterization

The leachate samples cannot be kept for long time periods because of active anaerobic activity. So the leachate samples were collected only after the laboratory was set up and ready. The leachate samples were collected from the monitoring well located in the center of the land fill. The screen of the well is at a depth of 4 m, within the waste body. A small portable submersible pump was used to collect the samples (Figure 4.9). The collection also included groundwater samples from wells in the vicinity of the monitoring well, both from locations upgradient and downgradient of the landfill. The location, pertinent detail and field data from these wells are included in Appendix B.

Groundwater and leachate samples were returned to the laboratory for chemical analysis; the results of these analyses are shown in Table 4.6.

As previously discussed, heavy metal content of the leachate is an important factor in the resistivity measurements. Concentration of inorganic species in leachate is shown in Table 4.6. Values for total hardness (CaCO_3 content), TDS and conductivity values of the leachate were 213.9 mg/l, 17,700 mg/l and 20,740 $\mu\text{S}/\text{cm}$, respectively.



Figure 4.9 Groundwater sampling at the monitoring well.

Table 4.6 Chemical analyses of the leachate and groundwater sample from the study area.

Chemical analysis	MW-MH-8 (Upgradient)	MW-MH- L2 (Leachate)	MW-MH-3uw (Downgradient)
Color Pt-Co unit	5	-	5
Turbidity (NTU)	1.1	-	0.6
Carbonate CO_3^{2-} (mg/l)	0.0	0.0	0.0
Bicarbonate HCO_3^- (mg/l)	317	-	464
Total alkalinity CaCO_3 (mg/l)	260	-	380
Total hardness CaCO_3 (mg/l)	246.4	213.9	366.2
TDS (mg/l)	448	17,700	1,900
Conductivity ($\mu\text{S}/\text{cm}$)	526	20,740	2,254
Na (mg/l)	40.44	419.45	338.363
K (mg/l)	7.98	627.56	12.80
Ca (mg/l)	88.36	48.02	120.97
Mg (mg/l)	6.198	22.82	15.50
Fe (mg/l)	0.18	30.90	0.290
Mn (mg/l)	0.005	0.467	0.405
Pb (mg/l)	0.006	0.017	0.005
Cd (mg/l)	<0.002	0.004	0.002
Zn (mg/l)	0.050	0.791	0.042
Cu (mg/l)	0.007	0.188	0.011
Cl (mg/l)	16.1	-	572.8
F (mg/l)	0.2	0.4	1.0
SO_4^{2-} (mg/l)	10.5	-	5.2
NO_3^- (mg/l)	3.81	-	0.38
As (mg/l)	<0.005	<0.005	<0.005

The leachate fluid, being one of the most important materials in this project, was collected in large amounts from the field and stored in a refrigerator. For the experiment, the leachate was diluted in various concentrations with normal water. Leachate contains large quantities of total dissolved solids, with a specific gravity similar to normal groundwater. The leachate do not sink nor float but tended to dilute very well with groundwater. The dilution processes are depended on the diffusion and dispersion rate. It is difficult to estimate just how dilute the leachate becomes as it migrates away from the landfill and mixes with groundwater.

In this case, the higher concentration of leachate fluid is a better representative of contaminating circumstance. Therefore, three different concentrations of leachate solutions, 25%, 50%, and 100% were decided to use in the experiment. These concentrations should be sufficient to represent the contaminating conditions in the field.

In additional, there was an attempt to set up the experiment with 2.5 % leachate solution, rain water, fresh water and tab water, because these fluid might be possible saturating fluids in the field condition also. The 2.5% leachate represents the same conductivity value as normal hardness groundwater, and rain water was collected from the local represents the condition of in situ sediment during the rainy season, also tab water that might be saturated in the plantation area.

In order to use Archie's equation, the conductivity or resistivity of the pore fluid must be known. Therefore, the conductivity of the solutions used in this experiment was measured by using conductivity meter. The measurement also made (for comparison purposes) with the resistivity box. These measurements are shown in Table 4.7

The electrical resistivities of all three concentrations of leachate used in this experiment measured less than 2 ohm-m. Taylor and Baker (2006) determined that the Archie relationship is only valid over a small range of electrolyte resistivity (< 2 ohm-m). Thus this experiment is consistent with the Archie Equation.

Table 4.7 Conductivity and resistivity values of various fluid samples.

Type of fluid	Conductivity meter measurement		The resistivity box measurement (ohm-m)				
	Conductivity ($\mu\text{S.cm}^{-1}$)	Resistivity (ohm-m)	1 st set	2 nd set	3 rd set	4 th set	Average
Deionized water	2	5,000	7,544.30	6,989.42	7,100.69	7,094.19	7,182.15
Fresh water	46	217.39	371.05	369.34	355.18	354.39	362.49
Tab water	105	95.24	-	-	-	-	-
Rain water	56	178.57	-	-	-	-	-
100% leachate	22,600	0.44	0.46	0.44	0.45	0.45	0.45
50% leachate	11,600	0.86	0.69	0.74	0.70	0.74	0.72
25% leachate	6,100	1.64	1.37	1.37	1.41	1.45	1.40
2.5 % leachate	770	12.92	-	-	-	-	-

4.3 Experimental design

The procedures for measuring electrical resistance were as follow: First, soil, pure water and leachate were prepared, and the bulk density, porosity, water content, and saturation index of the four types of sediments were measure. The volume of the samples needed in experiment was calculated from these parameters, and the volume of leachate needed to make the sample reach its saturation index was also calculated. Second, the test sample consisting of soil and leachate solution was pored into the PVC container of the resistivity box and compacted to simulate natural conditions. The details of specimen compaction are described below in Section 4.3.1. Finally, the electrical measuring system was attached to the filled apparatus and the resistivity was measured. The variation of electrical resistivity was measured for each type of soil with changing degree of saturation and leachate concentration. All measurements were duplicated under both quantity and quality control. If the data for the duplicate samples were different by more than 10%, additional measurements were performed and the values were averaged.

4.3.1 Sample preparation

The soil samples taken from the field were unsealed and left to dry first at room temperature and then in an oven at 40° C. Oven dried the samples with the increasing temperature 10° C every 24 hours till the temperature reached a maximum 70° C. By slowly drying the samples in this way, they were completely dried and the organic matter and the pore spaces were well preserved. If the grains in the samples linked into clusters, ground it softly with a mortar and pestle.

Packed the sample into the resistivity box so that it simulated field conditions, by referencing the bulk density of that soil obtained from the previously-studied samples (see Section 4.1.2). The bulk density is the ratio of bulk volume of the soil to its mass. In this case, if a quantity of soil is packed inside the resistivity box, the bulk volume of soil must be equal to the box's volume. Thus the mass of the soil needed is its bulk density multiplied by the volume of the box. The sample was weighted carefully then packed the entire amount of sediment so that it fit perfectly into the entire volume of the box. If the fit was not perfect, shall took out the sample

and repack it. It is sufficient to measure the height of the packed sample since the volume of the soil is known from the initial weighing (Figure 4.10).

The leachate solution were then poured onto the top surface of the packed sample, closed the attached the lid, and connected the box to the vacuum pump (Figure 4.11) through the valve at the bottom, and turned on the pump for about 20 minutes, then allowing the sample to soak thoroughly. The time allowed for soaking depended on the type of sediment as follows: 20 minutes for sand, 2 hour for clayey sand, 6 hours for sandy clay, and 8 hours for clay. The total soaking time for each sample was estimated from the saturated hydraulic conductivity rates for different soil texture measured by INSTAAR (The Institute of Arctic and Alpine Research), 2002.

After the sample was allowed to soak for the appropriate period of time, do the electrical resistivity measurement of the sample with water content at certain saturation degree. More testing fluid was then added, for the sample to reach the next degree of saturation index. The resistivity measurement was then repeated. Additional leachate was added in small steps, started from the lowest degree of saturation (the permanent wilting point) until the saturation index equaled 1.0 and the sample was fully saturated.



Figure 4.10 Packed test specimens in the resistivity box.



Figure 4.11 Vacuum pump attached to the resistivity box's valve as a pressure control system of the experiment.

4.3.2 Measurement procedures

The experimental set up consists of four major components: the sample, the resistivity box with pressure control (a vacuum pump), the electrical measuring system, and the fluid flow system. The sample was described above, and the details of the resistivity box and electrical measuring system were described in Chapter 3.

The same set up for the resistivity experiments was followed as when the box was calibrated. After the resistivity box was packed with a sample, and the sample was allowed to saturate with leachate, the current electrodes of the resistivity box were applied using an AC power source, operating at 50 Hz at 5 volts. The current of the circuit was measured in terms of voltage over a series connecting a known resistor in the circuit. The voltage was then converted to ampere by using Ohm's law. Then the voltage measurement was done over the potential electrodes of the resistivity box with the packed sample. By replacing the pre-measurement current (I), and applying Ohm's law, the resistance of the sample inside the box can be calculated. Then the measured resistance was multiplied by the geometry factor obtained from the box's calibration.

This six-electrode resistivity box had four sets of electrodes in the Wenner configuration, so each measurement included four resistivity values. The average of

the four values was the representative value of the sample with a given degree of saturation.

Note that in order to measure the current of the measuring circuit it is necessary to measure in terms of voltage over a known resistor. But when the measurements are performed on samples which have a very high resistance, and the known resistor has a resistance that is very low compared to the sample, the voltage over the known resistor will be too big and the voltage over the resistivity box will be too low. The result is an error on the oscilloscope reading. If the value of known resistor is too high, then the total resistance of the circuit will be much too high so that the electricity cannot pass through the circuit, and nothing can be measured.

This, the resistance of the known resistor used in the circuit should be about 3 to 0.3 times that of the sample. More than twenty different ceramic resistors were spared, so it was able to choose the proper known resistors for the circuit no matter the resistivity of the sample. To find the appropriate known resistor, any random resistor was used in the first measurement, and then changed the known resistor into the proper range after considering the first result.

The four type of sediment were saturated with leachate solution of 0%, 2.5%, 25%, 50%, 100% concentrations, with the solution's conductivities measured at $0.2 \mu\text{S}\cdot\text{cm}^{-1}$, $770 \mu\text{S}\cdot\text{cm}^{-1}$, $6,100 \mu\text{S}\cdot\text{cm}^{-1}$, $11,600 \mu\text{S}\cdot\text{cm}^{-1}$ and $22,600 \mu\text{S}\cdot\text{cm}^{-1}$. The electrical resistivity measurements were done at a variety of degrees of saturation. As mentioning above, all tests were performed at least twice. If the data for the duplicate samples differed by more than 10%, additional measurements were performed and the average values were used.

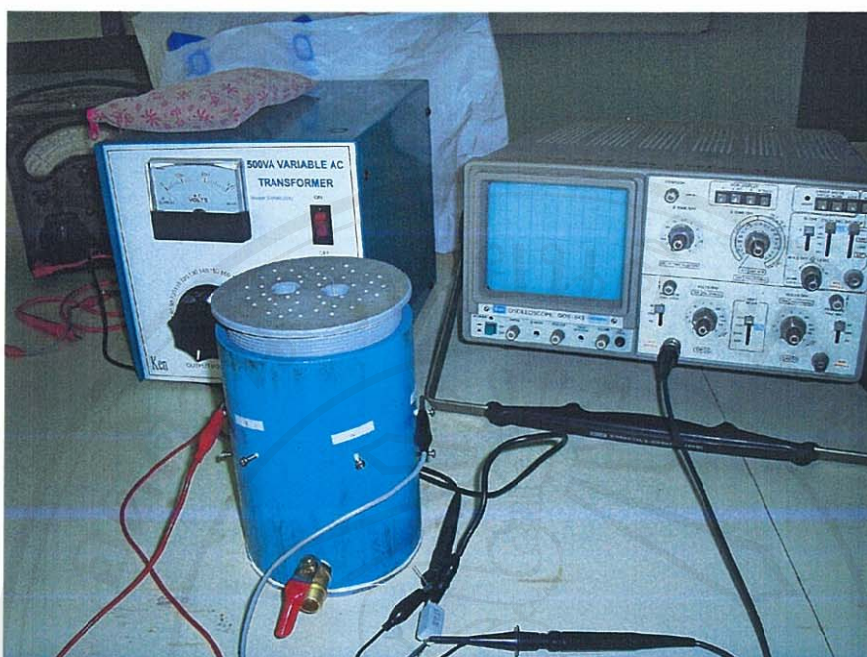


Figure 4.12 The experimental set-up.

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