APPLICATION OF COMPUTED TOMOGRAPHY FOR VISUALIZING THREE-DIMENSIONAL FABRIC AND MICROSTRUCTURE OF FRASER RIVER DELTA SILT

by

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The following individuals certify that they have read, and recommend to the Faculty of Graduate and Postdoctoral Studies for acceptance, the thesis entitled:

Application of computed tomography for visualizing three-dimensional fabric and microstructure of Fraser River Delta silt

submitted by Michelle Wesolowski in partial fulfilment of the requirements for the degree of Master of Applied Science in Civil Engineering

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Abstract

A research program was undertaken to visualize and quantify the three-dimensional nature of fabric and microstructure of natural silts with the objective of better understanding the influence of these factors on macroscopic monotonic and cyclic soil behaviour. The development of technology for the visualizations using X-ray micro-computed tomography (CT) formed a key task. Natural, low-plastic Fraser River Delta silt available from the Lower Mainland area of British Columbia was used as the geomaterial for the study. In order to capture a representative elemental volume for analysis in micro-CT, experimentation was undertaken to identify the appropriate method(s) to obtain sub-samples of specimens from relatively larger undisturbed samples or reconstituted specimens of silt.

Thin-walled (0.135-mm thick) plastic tubing (5.0-mm diameter) was chosen to obtain sub-samples of silt that would be compatible with micro-CT scanning apparatus and procedures. The potential for sample preservation using resin impregnation was also explored. A very low viscosity resin which cured at room temperature under anaerobic conditions provided a novel way to successfully preserve samples. Preliminary observations suggest that there is minimal disturbance to the internal fabric and microstructure within the core of the specimen sub-samples.

A collaboration with three X-ray micro-CT laboratories allowed for scanning of the silt sub-samples to voxel resolutions ranging from 0.869 to 3.38 µm. The three-dimensional datasets were then post-processed using commercially available software. A systematic study was conducted to choose the “non-local means” filter which reduced image noise while preserving digital grain edges. Particle segmentation of the images was undertaken using the watershed methodology, which led to successful digital grain size distribution matching with typical laboratory data. Initial quantitative analysis indicates that the void ratio as well as particle contact angle distribution diagrams can be formulated for silt-sized material. Quantification of particle shape including sphericity, roundness, and aspect ratio, and their relation to specimen mineralogy, was also explored. The research work demonstrated that X-ray micro-CT technology has a strong potential to be a viable method for three-dimensional visualization of silts.
Lay Summary

Various soils display different results for strength and stiffness depending on how the specimens are prepared for laboratory testing. Soils retrieved directly from the field with little disturbance seem to show results that are different from those obtained from laboratory-prepared specimens.

These observed differences in soil behaviour can be attributed partially to the physical arrangement of particles (fabric) and microstructure of the tested soils. Research in quantifying these phenomena specifically is very limited; especially with respect to soils such as silts that have small particle sizes. A good understanding of the mechanical behaviour of silts is critically important for geotechnical engineering design. This thesis presents a new methodology for preparing silt specimens for three-dimensional, non-destructive X-ray micro-computed tomography, proving that there is potential for this technology to qualitatively and quantitatively assess the microscopic fabric and structure of silts.
Preface

This thesis contains details of the research program undertaken at the University of British Columbia (UBC) Vancouver campus from September 2017 to December 2019. I, Michelle Wesolowski, along with assistance from Ana Valverde, was involved in the preparation of soil specimens for the purpose of sub-sampling for X-ray micro-CT analysis. I personally conducted all experimentation involving resin impregnation in the Civil Environmental Lab at UBC Vancouver. X-ray diffraction (XRD) analysis was conducted through the in the Department of Earth, Ocean & Atmospheric Sciences at UBC Vancouver. X-ray micro-CT scans were conducted by technicians at the three following locations:

- Geomechanics Laboratory, Department of Civil Engineering, Monash University, Australia
- Composites Research Network, Okanagan Laboratory, UBC Okanagan, Kelowna
- Pulp and Paper Center, Chemical and Biological Engineering, UBC Vancouver

I also performed all image processing in Avizo 9.7 and generated the code used for quantitative data analysis in MATLAB. All laboratory and computational experimentation were conducted under the supervision of Dr. Wijewickreme, who also reviewed and provided technical and editorial input for completion of the final manuscript.

Acknowledgement of respective authors for reproduced figures and data is given throughout the dissertation. Permission was given for presenting data gathered at UBC by Mavi Sanin, Achala Soysa, and Priyesh Verma. All other figures and data presented were prepared by the current author.
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My grandparents Halina and Wladyslaw Rabczak and Wanda and Jan Wesolowski:
for their powerful and loving presence in my life; together or apart.
1.0 Introduction

1.1 Background

Based on experience from many past earthquakes, saturated loose sands have been well identified as susceptible to liquefaction; more recently, cases such as the 1991 Chi-Chi, 1999 Kocaeli, and 2011 Christchurch earthquakes (Bray et al. 2004; Idriss & Boulanger, 2010; Cubrinovski et al. 2019) have shown that silty soils are also problematic soils in this respect. As such, specific focus has been placed on low-plastic silty soils in the Lower Mainland area of British Columbia. Fraser River Delta silts in particular have been the focus of research at the University of British Columbia for over 18 years. In this work, the monotonic and cyclic shear behaviour of relatively undisturbed and reconstituted slurry deposited silts has been investigated using a variety of methods including laboratory direct simple shear and triaxial testing. Several factors, including effective confining stress, over-consolidation ratio, coarse-grained fraction, initial static shear bias, soil plasticity, and fabric and microstructure, have been shown to affect the behaviour of silts (Wijewickreme et al. 2019).

A breadth of past soil mechanics and geotechnical engineering research has shown that the fabric and microstructure (e.g., differences in the behaviours between the results from testing of undisturbed versus reconstituted soils; variation of fabric due to the method of reconstitution during laboratory element testing) has a marked effect on the macroscopic monotonic and cyclic behaviour soils (Mulilis et al. 1977; Miura & Toki, 1982; Zlatovic & Ishihara, 1997; Vaid et al. 1999; Høeg et al. 2000; Wood et al. 2008; Sanin, 2010; Soysa, 2015). Research addressing this regime specifically with respect to silt has been limited (Wijewickreme et al. 2019). In addition to this, many researchers have demonstrated that in sands, significant particle reorientation occurs during consolidation, notably that the long axis of the particle tends to align itself more perpendicular to the applied load as the load increases (Paniagua et al. 2015). Observations of particle sphericity and aspect ratio have also known to cause varying changes in sample fabric and microstructure (Yang et al. 2019). The true microscopic
fabric and structure of undisturbed silt specimens compared to slurry deposited silt
specimens has never been explicitly explored.

The above has demonstrated that there is a lack of investigation in fabric and
microstructure of low-plastic silt material. Due to its potential risk for liquefaction, it is
imperative to understand how varying laboratory practices on undisturbed and
reconstituted specimens affect the soil’s macro-behaviour, and how this behaviour is
translatable to the in-situ field strength.

Representative quantification of fabric and microstructure of fine-grained materials has
been limited due to many constraints – including the difficulties in not disturbing the
fabric and microstructure during sampling and specimen preparation. Optical
microscopy and scanning electron microscope (SEM) technologies have been used in
the past, however these methods are restricted to a two-dimensional plane of viewing
(Reyes et al. 2017). Fabric and microstructure are inherently three-dimensional due to
the complex shapes, arrangements, and orientations of particles and voids. X-ray micro-
computed tomography has been successfully used in the past for the non-destructive
visualization of sand particles (above 75 µm in diameter) and voids (Ketcham &
Carlson, 2001; Taina et al. 2008; Cnudde & Boone, 2013; Helliwell et al. 2013), as well
as a variety of other materials including concrete, rock, additive metals, and pulp. The
ability to capture a representative reconstruction of full, individual particles is restricted
by the scanning resolution, or voxel size.

Recent developments in X-ray micro-computed tomography technology, accompanied
by increased computing power capabilities, have shown that imaging of silt-sized
particles ranging from 2 to 75 µm in diameter is now possible (Zhang & Jivkov, 2016;
Markussen et al. 2019). Research specifically on silt for the application to geotechnical
engineering is extremely limited to non-existent.

Previous two-dimensional imaging techniques have failed to capture the undisturbed
specimen fabric and microstructure which is contributing to the silt behaviour, further
reaffirming the need for X-ray micro-computed tomography analysis.
1.2 Objectives

The main objective of this thesis is to develop a methodology for capturing the three-dimensional fabric and microstructure of Fraser River Delta silt specimens. There is currently no simple best-practice process for preparing, imaging, and processing silt specimens with the intention of analyzing their particle characteristics and influence on monotonic and cyclic shear behaviour. In consideration of this, the below is a list of goals that were set for the purpose of this research:

- Develop a sampling method (including sampling tube type) that would be appropriate for X-ray micro-computed tomography scanning of silts, while imparting minimal disturbance to the specimen fabric and microstructure during such sampling
- Investigate the possibility for preserving (sometimes also referred to as “fixing”) the specimens via methods of very low viscosity resin impregnation
- Determine the necessary imaging devices and scanning parameters required to obtain images of resolutions greater than or close to the minimum particle size of the silt specimens
- Digitally capture individual silt grains in order to analyze the sample’s grain size distribution, void ratio, particle shapes, and primary axis orientation (rose diagram) and correlate the aforementioned characteristics to the specimen’s observed laboratory behaviour
- Observe the differences in fabric and microstructure between undisturbed and slurry deposited reconstituted specimens as well as specimens consolidated to various stress levels
- Analyze specimen mineralogical composition to evaluate the influence of mineralogy on silt behaviour, fabric, and microstructure.

If the above were to be successful, a methodology could be developed for classifying silt behaviour to its fabric and microstructure. Eventually, samples could be extracted from the field, preserved, and sent immediately to the X-ray micro-computed tomography scanner for analysis which would reveal preliminary estimates of soil behaviour based on the observed fabric and microstructure.
1.3 General Thesis Outline

This thesis is comprised of six chapters. Chapter 1.0 introduces the research question by drawing attention to the need for assessment of fabric and microstructure of silt material. It highlights that X-ray micro-computed tomography is the next frontier for non-destructive three-dimensional imaging in this regard. Chapter 2.0 is a detailed review of literature and past research relevant to the topic of monotonic and cyclic behaviour of silt, and the influence of the method of reconstitution on soil behaviour. It also reviews the definitions of fabric and structure, as well as several aspects of particle shape. Sample preservation methods, imaging techniques for geomaterials, and image processing software are presented. Chapter 3.0 encompasses the laboratory experimental aspect of this research. It outlines the material tested, the systematic study of sampling tube shape and material, specimen preparation, and experimental resin impregnation methods. It also summarizes the testing program, including all of the specimen types that were prepared for imaging. Chapter 4.0 discusses all aspects relating to X-ray micro-computed tomographic imaging as well as the computational post-processing methods used for particle segmentation. Chapter 5.0 presents results and discussion of various imaged specimens, as well as demonstrates the capabilities of the software and post-processing methodology for capturing a representative digital reconstruction of the laboratory specimens. A summary and conclusion of final results, in conjunction with recommendations for future research are made in Chapter 6.0, followed by references and appendices.
2.0 Literature Review

Findings from over 18 years of research at the University of British Columbia (UBC) indicates that the that the particle fabric and microstructure have a strong influence on the macroscopic, mechanical behaviour of Fraser River Delta silt (Wijewickreme & Sanin, 2004; Sanin & Wijewickreme, 2006; Sanin, 2010; Soysa, 2015; Verma, 2019; Wijewickreme et al. 2019). The background from this mechanical understanding has been the motivation behind this research for observing and analyzing the three-dimensional particle fabric and structure at the microscopic level.

The literature review presented in this chapter introduces the considerations relating to particle fabric and structure and its applications to silt behaviour. Section 2.1 defines fabric and structure and how they pertain to soil research. Section 2.2 reviews methods of specimen extraction for laboratory testing, and how the specimen extraction process may cause disturbance of soil fabric and microstructure. Section 2.3 covers typical stress-strain behaviour for monotonic and cyclic shear testing of undisturbed laboratory specimens; this section highlights that there is limited data for low-plastic silt research, particularly in relation to its liquefaction susceptibility that has further motivated the pursuance of this work. Section 2.4 reviews specimen reconstitution techniques and highlights the widespread influence that the chosen method has on resulting soil behaviour for both monotonic and cyclic testing. Section 2.5 discusses specimen preservation techniques such as resin impregnation that have been used in attempts to preserve soil architecture. Section 2.6 highlights imaging techniques that have been used in order to quantify soil fabric and structure and introduces X-ray micro-computed tomography as the next frontier for high-resolution particulate imaging. Finally, Section 2.7 discusses image processing software that have been used for the application of post-processing and particle segmentation for further quantification analysis.

2.1 Fabric and Structure

Soil researchers have long debated the distinction between soil fabric and structure. FitzPatrick (1984) reviewed several definitions of fabric and structure, comparing the Oxford English Dictionary definition to German soil scientist translations, dating back to
the 1930s. In their book on clay microstructure, Bennett and Hulbert (1986) make references to pioneering works by Terzaghi, Casagrande, Lambe, and Mitchell, highlighting that the concepts of fabric and structure have been a point of keen interest to geotechnical engineering applications for nearly a century.

Fabric, with respect to soils, is defined as the spatial arrangement of solid particles and voids. The voids in this sense are only those that are a direct result of the physical orientation and packing of the soil particles (Brewer & Sleeman, 1960). Fabric focuses primarily on the characteristics of individual particles such as shape and size, and how this has led to their particulate arrangement (FitzPatrick, 1984; Santamaria, 2001). Oda (1972) described that there are two main components to fabric, which are listed below and shown schematically in Figure 2.1.

- The particle’s discrete orientation, and
- Its relative position with respect to adjacent particles

![Figure 2.1 Definition of particle fabric (modified from Oda, 1972)](image)

Rose diagrams are common tools used to visualize the preferential orientation of particles in large data sets (Boggs Jr., 2006; Nichols, 2009; Fawad et al. 2010; Fonseca et al. 2016). The primary directional vector of each grain is plotted in a form of polar histogram to quantify changes or patterns in particle orientation. In order to quantify the relative configuration of particles, the coordination number (CN) is often used, which is
the number of adjacent particles that are in physical contact with a single particle. Particle contact area is also investigated. The aspect ratio \( A_R \) is used to describe shape and is defined as the ratio of the smallest diameter to the largest diameter of a particle (Equation 2.1). An \( A_R \) of 1.0 is equivalent to a sphere.

\[
A_R = \frac{d_{\text{min}}}{d_{\text{max}}}
\]  

Soil structure, in terms of general pedology, is defined not only as the arrangement of particles, but the entire interconnected soil system and its related pore spaces (FitzPatrick, 1993). This can include the fluid or gas that infills these pores, as well as the chemical or electrostatic bonding that may exist between individual soil particles (Craig, 1992; Chang et al. 2011). The definition of structure is distinctive, as it includes peds, or aggregations, that develop within soils (Brewer & Sleeman, 1960; FitzPatrick, 1984). Types of soil structure include single-grained, blocky, columnar, platy, and massive (Engle et al. 1991; Tremback, 2018). In his basic characterization of soils, Craig (1992) presents “single grain structure”, which is common in sandy soils. Soil structures are often classified in hydropedology and groundwater management by their ability to pass water (Engle et al. 1991). Single-grained structure has rapid flow, whereas blocky and columnar structure has moderate flow, and platy and massive structure has slow flow.

Clays can also display a wide range of structure types such as dispersed, card house, and deflocculated (Figure 2.2). Clays with a card house structure are often referred to as “quick clays” (Bennett & Hulbert, 1986), and can sometimes lead to collapse, such as in the case of Leda clay found in the Ottawa valley of Canada. Problem soils such as these showcase the need for research into soil structure to further understand their failure mechanisms. While this research will focus on silt-sized materials, it is important to recognize that soil specimens contain a wide range of particle sizes and fines, and therefore, these structures may be present under a given real-life situation.
Both soil fabric and structure are influenced by their genesis, or sedimentological history, however their distinction becomes more prevalent when referring to soil mechanics. In soil mechanics, the definition of fabric also encompasses the interparticle forces experienced at the grain contacts (Santamarina, 2001; Hight & Leroueil, 2003; Yamamuro et al. 2008, Fonseca et al. 2016). Microstructure of clays has also been classified by Hight and Leroueil (2003) as observing the void ratio, the strength, and the yield stress of an intact specimen as a whole.

This thesis is specifically concerned with silt-sized materials, defined here as particles passing the No. 200 sieve, ranging in size from 2 μm to 75 μm (ASTM D653-14, 2014). As such, specimen investigation occurs at the micro-scale (10⁻⁶). Therefore, the term “microstructure” will be used henceforth. It is important to make this distinction, as
particle physics theories behind behavioural mechanisms can be influenced by the scale of investigation (Hight & Leroueil, 2003; Mei, 2017). The above review demonstrates the complex link between fabric and microstructure. Depending on the scale of research and investigation, it may be necessary to analyze both components.

2.1.1 Effects from Particle Shape

Particle shape is often studied in order to draw conclusions on the soil’s past morphological and depositional history. Certain aggregations of particle types and textures may be indicative of depositional environment and energy. For example, aeolian deposits, such as sand dunes, often have very well-rounded, spherical soil particles. Glacial sediments may have striations due to scraping at the glacier base and be deposited with their long axis in the orientation of glacial movement. Particle shape can also be influenced by the mineralogical make-up of the source material.

A variety of classification methods have been developed in order to quantify characteristics such as roundness (conversely defined as angularity) and sphericity, all of which help describe the particle shape. Wadell (1932) first defined roundness by Equation 2.2, which considers the radius of curvature of various particle corners and the radius of the largest possible sphere (Figure 2.3). This definition is still used widely in sedimentology and pedology (Boggs Jr., 2006).

\[
Roundness = \frac{\sum_{i=1}^{n} \left( \frac{r_i}{R} \right)}{n}
\]  

(2.2)

Figure 2.3 Schematic of roundness, as per Waddell (1932)
Calculated sphericity ($\Psi$) is a function of the particle volume ($V_p$) and the smallest circumscribing sphere volume ($V_{cs}$) (Wadell, 1932). This is shown by Equation 2.3.

$$\Psi = \left( \frac{V_p}{V_{cs}} \right)^{1/3} \quad (2.3)$$

Sphericity can also be calculated by a particle’s long (a), intermediate (b), and short (c) axes, as shown by Equation 2.4 (Krumbein, 1941).

$$\Psi = \left( \frac{bc}{a^2} \right)^{1/3} \quad (2.4)$$

Shape classification schemes based on long, intermediate, and short axes have been developed by Zingg (1935) and Sneed and Folk (1958). An example of the Zingg (1935) classification is shown in Figure 2.4.

![Figure 2.4 Shape classification (modified from Zingg, 1935)](image)

Correlations to overall soil fabric and microstructure can be made to particular particle forms. For example, is the $D_s/D_L$ ratio is less than 0.3, and the sphericity is less than 0.7, then platy, bladed, and elongated particles can be expected, likely resulting in a platy structure. Santamarina and Cho (2004) were some of the first researchers to publish comprehensive findings on the influence of particle shape on soil behaviour and
force transfer between particles based on shape. Paniagua et al. (2015) further classified particles by their $A_R$, where particles with $A_R > 0.5$ were considered “bulky” while particles with $A_R < 0.5$ were termed “flaky”. They noted that flaky particles have a stronger tendency to reorient themselves perpendicular to the applied force. In a revealing study on sands by Yang et al. (2019), they report that the $A_R$ has a greater influence on the packing density of specimens, while the mechanical behaviour is more strongly governed by particle sphericity.

All of the above parameters mentioned are important to consider for overall specimen fabric and microstructure, as the shape has an effect on how the particles will pack themselves and behave mechanically. In order to observe these patterns, samples must first be extracted from the field, which is covered in Section 2.2.

2.2 Sample Extraction

Engineers conduct a wide range of laboratory tests such as Proctor testing, falling head testing, one-dimensional (1-D) consolidation testing, triaxial testing, and direct simple shear (DSS) testing (to name a few) to interpret geotechnical parameters such as soil compaction, hydraulic conductivity, rate of consolidation and settlement, and shear strength. Laboratory element testing plays an essential role in characterizing the behaviour of soils as a geomaterial.

In order to conduct laboratory tests, soils must be retrieved from the field in either an undisturbed or disturbed state and brought to the laboratory. Undisturbed sampling methods include block sampling and Shelby tube drilling. Grab samples and standard penetration test (SPT) split tube samples are considered disturbed, which means that their in-situ fabric and microstructure will most likely have been altered during the sampling process. While block and Shelby tube sampling may also slightly disturb the fabric and microstructure, these extraction methods are performed with great care and attention, and as such have been considered by industry to be the current best-practice of obtained relatively undisturbed samples.
2.2.1 Sampling Disturbance

Drilling methods such as Shelby/piston tube and block sampling have been established to minimize the sample disturbance, but these techniques are limited with respect to soil type and site material availability. Lunne et al. (1997, 2006), Ladd and DeGroot (2003), and DeGroot et al. (2005) have developed criteria for assessing sample quality that have been implemented for clays and soft ground characterization. Laboratory shear stress-strain behaviour obtained from specimens prepared from samples of lower quality have shown lower resistance at small strains compared to those from samples of higher quality (Lunne et al. 2006); as per this work, the normalized void ratio ($\Delta e_o/e_{\text{initial}}$) from sample storage to 1-D consolidation to the estimated in-situ conditions should be equal to or less than 0.07 for samples to be of “good to fair” quality. Block sampling has been identified as the method of least disturbance for soft clays (DeGroot et al. 2005); however, this method is not always feasible for soils of lower cohesion, such as silts.

Fabric and microstructure have also been referred to when analyzing the wall effects of drilling and sample extraction. Baligh et al. (1987) and Hight and Leroueil (2003) showed the strain induced on an in-situ sample using conventional piston sampling methods. The extent of strain is a function of the tube diameter and wall thickness, as shown in Figure 2.5.

![Figure 2.5 Disturbance due to sampler tube diameter and wall thickness (modified from Baligh, 1985 and Hight and Leroueil, 2003)]
Much research has been done on fine-grained soils to show the influence of disturbance from tube sampling due to increased porewater pressures and generated undrained shear response (Hvorslev, 1949; Clayton et al. 1998; Hight & Leroueil, 2003; DeGroot et al. 2005). Work by Lunne et al. (1997) compared shear stress response of specimens obtained via block sampling, 54-mm diameter sampling tubes, and 76-mm diameter sampling tubes. The peak shear strength obtained from block samples (which occurred at low strains), was nearly 50% greater than that of specimens obtained from 54-mm diameter tubing. The tests conducted specimens from 76-mm diameter tube samples performed marginally better than those from the 54-mm samples.

The above study demonstrated that there is a zone of disturbance that exists due to the sampling tube wall, and its effects are more evidence the smaller the inner diameter. Block sampling is not possible for low-plastic silt samples as they would likely slump and not retain their form without appropriate confinement; however, it is important to recognize and account for the effects of sampling disturbance when assessing soil behaviour.

In the following sections, observations from laboratory element testing on the general macroscopic mechanical behaviour of soils in general along with the effects of soil fabric on such behaviour are presented to serve as further background material.

2.3 Observations on the Macroscopic Mechanical Behaviour of Soils from Laboratory Element Testing

The macroscopic mechanical behaviour of soils has been studied through laboratory element testing by soil mechanics researchers over many decades. It has been well-established that the stress-strain response of soil is primarily governed by relative density (D_r) and effective confining stress (σ′_c). This has led to the development of Critical State Soil Mechanics (Schofield & Wroth, 1968) framework to characterize soil behaviour. The tendency to increase in volume (increase in void ratio, e) during shear is known as a “dilative” response, while a “contractive” response is when the soil decreases in volume (decrease in e).
This basis for macroscopic shear behaviour has been observed for both drained and undrained laboratory element testing. The drained condition is imposed in the laboratory to simulate loading cases where volume change could occur with less opportunity for shear-induced excess pore water pressures ($\Delta u$) to manifest – e.g., loading of coarse-grained soils that have relatively high permeability. On the other hand, the undrained condition is imposed to simulate conditions where the loading rate is such that dissipation of $\Delta u$ cannot take place to develop drained conditions – e.g., fine-grained material under relatively rapid loading such as excavations, or earthquake loading of coarse-grained soils. As silt-sized material is being studied for this research, the following section focuses on the results from tests conducted under laboratory undrained conditions; in particular, typical monotonic shear and cyclic behaviour is presented herein.

### 2.3.1 Typical Monotonic Shear Behaviour

Mainly via element testing of sand, there are three notable shear behaviour types that are well established in geotechnical soils testing (Vaid & Chern, 1985). Type 1 behaviour is known as the “contractive” response, where the sand develops shear-induced pore water pressures and in turn, displays a stress-strain response with a peak shear strength followed by “strain-softening” (or a decrease in shear strength with increased strain). This type of behaviour is classically described as liquefaction. Type 2 behaviour occurs when the sand reaches a peak shear strength, undergoes some strain-softening, then shows a dilative or strain-hardening response, which is when the shear strength increases along with strain. Type 2 behaviour is classified as a limited liquefaction case. Type 3 behaviour is dilative, where the sand continuously undergoes strain-hardening with increasing strain. This type of behaviour is considered stable (Chern, 1985; Kuerbis, 1989). The typical deviatoric stress versus axial strain curves and effective stress paths for monotonic undrained triaxial tests are shown in Figure 2.6.
Constant volume monotonic DSS tests on undisturbed Fraser River Delta silt specimens at various vertical effective consolidation stress levels ($\sigma'_{vc}$) were performed by Sanin and Wijewickreme (2006) and Soysa and Wijewickreme (2015). The results are presented in Figure 2.7. It can be seen that in the results for tests conducted in the $\sigma'_{vc}$ range of 100 – 500 kPa, the undisturbed soil specimens exhibit contractive behaviour initially, followed by a dilative response when observing the stress path diagram (Figure 2.7i). In Figure 2.7ii, the low-plastic, normally consolidated silt behaved contractively.
2.3.2 Typical Cyclic Shear Behaviour

Cyclic shear testing is performed in order to evaluate the resistance of soils to earthquake loading and assessing liquefaction potential. Liquefaction in the field can lead to many geotechnical hazards such as sand boils, flow slides, loss of bearing capacity, and uplifting of buried infrastructure. Lateral spreading and ground settlements have been noted extensively, as exemplified in the 1964 Alaska, 1964 Niigata, 1995 Kobe, 1999 Kocaeli, 2011 Christchurch, 2011 Tohuko, and 2015 Nepal earthquakes (Ishihara et al. 1997; Bray et al. 2004; Donahue et al. 2007; Seid-Karbasi & Byrne, 2007; Cubrinovski et al. 2019).

A significant portion of liquefaction research has been performed on saturated, clean sands, as historically these were the soils that were encountered in liquefaction cases. Most experimental studies however have been conducted on reconstituted specimens due to the difficulties in obtaining high-quality undisturbed sand samples for laboratory
testing. Clays have been largely accepted as non-liquefiable, in part due to their electrochemical bonding structure. Silts of low-plasticity however, have displayed susceptibility to liquefaction, as noted from 1991 Chi-Chi, 1999 Kocaeli, and 2011 Christchurch earthquakes (Bray et al. 2004; Idriss & Boulanger, 2010; Cubrinovski et al. 2019). Research in recent years has shown that silt liquefaction is of keen interest, especially in coastal regions of high seismicity such as Vancouver, California, Japan, and New Zealand.

The phenomena of liquefaction due to cyclic loading is interpreted with respect to a reduction in shear stiffness and shear strength due to an increased in shear-induced excess pore water pressure. Quantitatively, it has been defined in several ways by many researchers. For cyclic DSS testing, 3.75% single-amplitude shear strain is used as an index for liquefaction triggering (NRC, 1985; Wijewickreme & Sanin, 2004; Soysa, 2015) mainly to serve as a basis for comparing data and establishing cyclic resistance ratio (CRR) curves for design. The greater the cyclic resistance of the soil, the greater the number of cycles required to reach the CRR.

Typical effective stress path and shear-strain response for a contractive (loose) material and dilative (dense) material is shown in Figure 2.8i and Figure 2.8ii, respectively. In the case of the loose material (Figure 2.8i), in the initial cycles of loading, there is only a small accumulation of shear strain with a relatively high shear stiffness; this is followed by an abrupt degradation of the shear stiffness almost over the duration of one load cycle. This strain development has been referred to as liquefaction (Vaid & Chern, 1983). In Figure 2.8ii, a typical shear response for a relatively dense material is shown. Herein, the shear stiffness is gradually reduced with increasing cycles, and as such, the soil does not experience an abrupt degradation stiffness. This strain development is referred to as “cyclic mobility”.
Figure 2.8 Typical stress-strain response of (i) contractive (loose) sand showing liquefaction (data from Wijewickreme et al. 2005) and (ii) dilative (dense) silt showing cyclic mobility (data from Sanin, 2010)

Results from Sanin (2010) in Figure 2.9 shows the cyclic mobility of an undisturbed Fraser River Delta silt at $\sigma_{vc}' = 100$ kPa reaching 143 cycles at 3.75% strain. Other undrained monotonic DSS cycling loading tests by Dahl et al. (2014) of Potrero Canyon, California undisturbed low-plasticity silty sand (ML) of PI = 6 also show cyclic mobility behaviour. Zhang et al (2017) presented undrained cyclic triaxial tests on silt material of PI = 8.8 in the Tianshui area of China known for landslides and dynamic liquefaction susceptibility. All specimens of $\sigma_{vc}'$ ranging from 100 – 800 kPa showed typical cyclic mobility.

Figure 2.9 (i) Stress-strain and (ii) stress path results of constant volume cyclic DSS test on undisturbed Fraser River Delta silt showing cyclic mobility (data from Sanin, 2010)
All of the above soils were relatively normally consolidated, undisturbed specimens. As mentioned previously, undisturbed field samples are often difficult to obtain, and cyclic loading and liquefaction assessment of undisturbed silts is very limited. Silt behavioural testing is often performed on reconstituted specimens to examine the influence of factors such as fabric, sand-silt mixture effects, presence of fines, plasticity, mechanical overconsolidation ratio (OCR), confining stress, and static shear bias, to name a few. Specimen reconstitution, and the resultant monotonic and cyclic soil behaviour, is discussed in detail in Section 2.4.

2.4 Evidence of Microscopic Soil Particle Fabric Effects from Laboratory Element Testing

Evidence linking the macroscopic behaviour, as covered in Section 2.3, to the microscopic fabric and structure observed in laboratory prepared soil specimens is presented in this section. Over 18 years of laboratory element testing by Wijewickreme et al. (2019) have shown that the method of reconstitution has a great deal of influence on the mechanical behaviour of Fraser River Delta deposits, both in the monotonic and cyclic condition.

This section summarizes several common reconstitution methods that have been developed for sand and silt testing and identifies the ideal method of reconstitution for undrained testing of alluvial silt sediments. It also covers findings from various studies showing the differences in monotonic and cyclic behaviour of soils due to reconstitution, with a focus on silt materials.

2.4.1 Types of Laboratory Reconstitution Methods

When performing laboratory testing on a disturbed soil sample, a specimen must be reconstituted, or remolded, in order to conform to a mold as specified by standard testing procedures. The chosen method of reconstitution is reliant upon several factors, including:

- Soil type: gravel, sand, silt, clay, etc.
- Depositional history: alluvial, fluvial, aeolian, etc.
• Test type: drained or undrained
• Relative density: low, moderate, high
• Intended soil construction method: level of compaction, hydraulic fill, etc.
• Other parameters under investigation: water content, void ratio, etc.

Depending on the intended application, soil can be deposited in a dry or wet condition. Air pluviation and water pluviation are common techniques used for sands (Mahmood et al. 1976; Kuerbis & Vaid, 1988; Wijewickreme et al. 2005).

In air pluviation, dry sand is deposited from a volumetric flask at a specified drop height depending on the desired density. This technique is favourable for poorly graded sands, as segregation may occur during drop height. Vibratory methods can be used to increase specimen density, however further segregation of heavier, larger particles to the bottom may occur. Additionally, when the specimen is saturated, there is risk of fabric disturbance and fines washing out. This method is considered for aeolian-type sand deposits as well as in fundamental studies on the behavior of sands.

Water pluviation can be used for reconstitution of sand. The method is similar to air pluviation; however, the soil is deposited underwater. This allows particle flocculation, producing a more natural deposition for sediments of alluvial or fluvial origin (Thomson & Wong, 2008). Turbulence can be caused due to a single depositional spout, but researchers use various techniques to overcome this. Segregation can also occur due to finer particles remaining suspended for longer. Water pluviation is generally not suitable for silts as settling time can be significant, and the resulting specimens can be very loose and soupy, even after decanting.

Moist tamping involves depositing and densifying specimens in layers to a desirable height. Layers are tamped based on predetermined ratios of the soil’s $e_{\text{min}}$ and $e_{\text{max}}$ as well as energy produced from drop height. Highly dense specimens can be achieved using this method. To achieve a more uniform fabric, the bottom layers are under-compacted and the top layers over-compacted so that the center layer achieves the target void ratio (Ladd, 1978; Frost & Park, 2003). Chang et al. (2011) note that particle orientation is more random using the moist tamping method, which may not be
representative of certain sedimentation environments where flocculated particles are deposited parallel to their long axis. This method may not be achievable for soils that are predominantly silt as the $e_{\text{min}}$ and $e_{\text{max}}$ cannot be accurately determined using traditional ASTM methods (Bradshaw & Baxter, 2007).

Slurry deposition has been a widely accepted method for silt reconstitution, particularly of silts deposited in the Fraser River Delta (Sanin, 2010; Soysa, 2015; Wijewickreme et al. 2019; Verma, 2019). After the soil is left to fully saturate under vacuum for about 24 hours, it is carefully spooned or funneled into the testing mold. Loose to moderately dense specimens can be achieved using this method, and since the soil is deposited in a highly wet state, back-saturation prior to testing is not required (Wang et al. 2011). This fully saturated state allows for platy particles to settle more naturally, and as such, the slurry deposition method has been deemed to be the most appropriate reconstitution method for alluvial deposits (Bradshaw & Baxter, 2007).

2.4.2 Influence of Specimen Reconstitution Method – Overview

Evidence has overwhelmingly shown that the method of reconstitution results in a wide range of behavioural variability, for both monotonic and cyclic testing. Table 2.1 outlines research programs of note that have been performed on soil reconstitution over several decades. The following two sections summarize findings on the influence of reconstitution on static and cyclic soil behaviour.
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<th>Reconstitution Method</th>
<th>Tests Performed</th>
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<tr>
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<tr>
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<td>• Slurry deposition</td>
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<td>• Water sedimentation</td>
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<td>• Isotropic consolidation</td>
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<td>• Undrained triaxial compression</td>
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<td>Thomson &amp; Wong (2008)</td>
<td>• Ottawa sand C778</td>
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<td></td>
<td>• Moist tamping</td>
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<td></td>
<td>• Water pluviation</td>
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<td></td>
<td>• Undrained triaxial compression and extension</td>
<td></td>
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<tr>
<td>Chang et al. (2011)</td>
<td>• ERPM Dam 4 gold tailings (Johannesburg, South Africa)</td>
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<td></td>
<td>• Sandy silt</td>
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<td>• Clayey silt</td>
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<td>• Moist tamping</td>
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<td>• Slurry deposition</td>
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<td></td>
<td>• Undrained triaxial</td>
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2.4.3 Influence of Specimen Reconstitution Method – Monotonic Behaviour

Researchers have attempted to compare soil behaviours arising from specimens prepared to identical initial void ratio and confining stress levels ($\sigma'_c$) using different methods of reconstitution.

Zlatovic & Ishihara (1997) presents data from undrained monotonic triaxial testing of Lagunillas sandy silt specimens normalized with respect to initial confining pressure. The moist placement and dry deposition methods yielded contractive strain-softening and contractive strain-hardening behaviour, respectively, while the water-sedimented specimen behaved dilatively. It was noted that while all three specimens reached similar low-strain peak shear strengths, their large-strain behaviours were significantly dependent on the method on reconstitution.

Static undrained simple shear testing on Syncrude sands at $\sigma'_v = 200$ kPa by Vaid et al. (1999) showed that with increasing axial strain, both moist tamped and air pluviated specimens demonstrate strain-softening contractive behaviour, while the water pluviated specimens behaved in a dilative fashion (Figure 2.10).

![Figure 2.10](image)

**Figure 2.10** Highly variable undrained simple shear response due to various reconstitution methods (modified from Vaid et al. 1999)
Høeg et al. (2000) compared the results from test conducted on moist tamped silty sand specimens with those from relatively undisturbed samples in monotonic undrained triaxial test at various confining stress states (low – 50 kPa, moderate – 250 kPa, and high – 500 kPa). The moist tamped specimens were prepared to nearly identical void ratios and moisture contents as their counterpart undisturbed specimens. The difference in behaviour between the undisturbed and reconstituted specimens becomes more defined with increasing confining stress. At 50 kPa, both specimens dilated. At 500 kPa, the moist tamped specimens contracted while the undisturbed specimens dilated and reached peak strengths up to 3 times higher than the reconstituted specimens.

Yamamuro and Wood (2004) and Wood et al. (2008) showed varied silt content in Nevada sand specimens for testing air pluviation, slurry deposition, water sedimentation, tapped funnel deposition, and fast funnel deposition specimens in undrained triaxial compression. For specimens of moderate density ($D_r \sim 54 – 60\%$), both the tapped and fast funnel deposited specimens showed greater strain-softening than the water sedimentation specimen (Figure 2.11). When the silt content was increased from 18% to 40%, this difference was even more significant. While only the fast funnel deposited specimen exhibited temporary liquefaction (contractive behaviour) at 18% silt content, both the tapped and fast funnel deposited specimens displayed this behaviour at 40% silt content. The water sedimentation specimen dilated with increasing axial strain for both silt content scenarios. This research led to a detailed review of various reconstitution methods, silt contents, and their shear-stress behaviour, which is summarized in Figure 2.12.
Figure 2.11 Further justification of variable stress-strain response of undrained triaxial specimens prepared via different reconstitution methods (modified from Yamamuro and Wood, 2004)

Figure 2.12 Volumetric behavioural trends based on reconstitution method and silt content (modified from Wood et al. 2008)
Northcutt and Wijewickreme (2013), using the findings from 1-D consolidation tests on sands, have suggested that it would be useful to consider a “fabric factor” when testing reconstituted specimens.

Chang et al. (2011) performed tests on tailings materials, and like Wood et al. (2008), with varying non-plastic fines content. Marked differences in behaviour were observed for moist tamped, slurry deposited, and undisturbed specimens through monotonic undrained triaxial shear testing. Overall, a 14% reduction in small strain stiffness was noted for moist tamped specimens compared to their undisturbed counterparts. For silt specimens at 200 and 400 kPa initial confining stress, the slurry deposited stress path more closely matched that of the undisturbed specimen, which behaved dilatively. The moist tamped specimens showed more strain-softening and tendency for contraction. For sand specimens, the moist tamped results were more agreeable with the undisturbed results, although both moist tamped and slurry deposited specimens showed greater strain-softening than the undisturbed.

Sanin (2010) showed that reconstituted Fraser River Delta silt specimens prepared using slurry deposition exhibited lower shear strength at all levels of confinement compared to undisturbed specimens (Figure 2.13). At larger strains, undisturbed specimens showed stronger Type 2 (contractive) tendencies. Similar works with silts of cyclic liquefaction susceptibility have been performed by Wang and Luna (2012), Dahl et al. (2014), and Zhang et al. (2017). They have all confirmed that the slurry reconstitution method can greatly affect the monotonic macroscopic behaviour of silts, thus inspiring further work showing its effects in cyclic testing which is discussed below.
2.4.4 Influence of Sample Reconstitution Method – Cyclic Behaviour

Generally speaking, under a given effective confining pressure, the stiffness and strength of soils are expected to increase with increasing relative density ($D_r$) due to their denser, more compact state.

Kokusho (2016) has summarized notable differences in cyclic behaviour of undisturbed and reconstituted silt specimens, despite maintaining the same relative density between specimens. Mullilis et al. (1977) showed that for clean sands at a $D_r$ of 50% and $\sigma'_c$ of 55 kPa, the cyclic stress ratio (CSR = $\tau_{cyc} / \sigma'_{v0}$, where $\tau_{cyc}$ = cyclic shear stress, and $\sigma'_{v0}$ = initial effective confining stress at a given point) of moist specimens prepared using a high frequency vibration method are almost double that of air pluviated specimens (Figure 2.14). Miura and Toki (1982) compared their multiple sieve pluviation reconstitution method to wet rodding for Toyoura sand. For a CSR of 0.2, the number of cycles to initiate liquefaction for multiple sieving pluviation and wet rodding were 4 and 20, respectively, at $D_r = 55\%$ and $\sigma'_c = 200$ kPa.
Figure 2.14 Cyclic stress ratio versus number of cycles for reconstitution of triaxial specimens on clean sand at consistent relative density (modified from Mulilis et al. 1977)

Cyclic DSS testing by Wijewickreme and Sanin (2008) and Soysa (2015) have revealed that reconstituted specimens show greater stiffness degradation with increasing number of load cycles compared to undisturbed silt specimens, in spite of the fact that former had post-consolidation void ratios smaller than the latter. For example, Figure 2.15 shows that by the 8th cycle, the shear strain accumulation is much greater for the reconstituted specimen, and the vertical effective stress is nearly half. As shown in Figure 2.16, the Site B cyclic resistance ratio (CRR, defined by the number of cycles to reach a single amplitude shear strain ($\gamma$) of 3.75\% ) is reduced by 14\% from 0.175 for the undisturbed specimen to 0.150 for the slurry reconstituted specimen when compared at 10 number of cycles.
Figure 2.15 Constant volume cyclic DSS shear stress-strain results of undisturbed and slurry reconstituted low-plastic Fraser River Delta silt specimens (data from Soysa, 2015)

Figure 2.16 Results showing cyclic stress ratio and number of cycles of undisturbed and reconstituted Fraser River Delta silt specimens demonstrating a reduction of cyclic shear resistance in reconstituted specimens (data from Wijewickreme and Sanin, 2008 and Soysa, 2015)
The significant differences above in the behavior patterns arising from different reconstitution methods, as well as between the undisturbed and reconstituted specimens, cannot be explained using the well-accepted critical state soil mechanics framework, which is founded on the basis that the void ratio and confining stress are the key parameters that would govern the stress-strain response of a given soil.

Based on this, it reasonable to state that the particle fabric and microstructure, would be the other major factor influencing the macroscopic monotonic and cyclic mechanical behaviour of soils, and silts particularly with regard to this thesis. However, the current understanding of the fabric and microstructure of silts (and in turn, the reasons for the varying mechanical behavior of the material) is very limited. This is due to the lack of research explorations conducted on this topic. In essence, there is a need to further study the effect of particle fabric and microstructure on the silt response, in addition to the traditionally well studied effects of void ratio \( (e) \) and \( \sigma'_{vc} \).

Research summarized in Wijewickreme et al. (2019) has derived notable conclusions on fabric and microstructure in the above regard. No studies have been done particularly focusing on the microstructure of silt-sized particle matrices. The currently available sophisticated imaging technologies should provide a way to understand particle structure of silts and assist in explaining the behaviours observed from mechanical element testing. For example, high resolution X-ray micro-computed tomography has been effectively used in visualizing grain fabric and obtaining microstructure of fine-grained materials for various applications. The process includes quantifying individual crystals or other discrete objects or void spaces, location, size, shape, orientation, and contact relationships with adjacent objects (Ketcham, 2005). Only a few studies have been conducted to image fabric and microstructure of fine-grained materials (Hain & Wriggers, 2008) and fiber reinforced polymers (Zhang & Jivkov, 2016).

In recognition of the above, a detailed research initiative was undertaken to examine the particle size, shape, and micro-structural arrangement of silt in different fabrics using imaging techniques. It was identified that several key technology development tasks as
given below need to be initially completed to launch this research work, thus forming the primary scope of work for the present thesis:

1. Preparation of silt specimens for fabric/microstructure imaging (e.g., resin-impregnation procedures for “fixing” silt matrix specimens for this imaging);
2. Identification of suitable scanning methods for fabric/microstructure imaging of silts, including equipment parameters for fabric/microstructure imaging;
3. Identification of suitable algorithms/software for post-processing of data collected from imaging; and
4. Verification of the chosen methodology.

Relevant literature related to these aspects are presented in the following section.

2.5 Specimen Preservation Methods

As noted in Section 2.4, the chosen method of reconstitution has considerable effect on the resulting fabric and microstructure. Researchers have been pursuing to visually capture this difference in microstructure by “fixing” soil specimens in their natural state through methods of preservation, including freezing and resin impregnation. The task of specimen preservation is performed in a wide variety of disciplines for the purpose of analyzing undisturbed characteristics, such as biology, botany, metallurgy, forestry, dendrology (wood studies), agriculture, and pedology. Resin impregnation has been implemented widely in the study of rock and coarse-grained sediments.

Sample freezing, both in-situ and in the lab, has also been done for specimen preservation (Seed et al. 1985; Scanziani et al. 2017). This method may be ideal for capturing only the macroscopic/overall soil fabric and structure, as shown by Liernur et al. (2017) on clay deposits. Additionally, the feasibility of keeping a sample 100% frozen is not easy and would require specialized transportation and storage methods. The increase in volume due to ice development in the void space causes changes in density, which irreversibly alters the fabric and microstructure. Freezing with the intention of preservation for scanning electron microscope (SEM) imaging analysis is also not possible as these samples must be ground and polished according to
specifications prior to imaging (Jang et al., 1999), unless using specialized cryo-SEM techniques (Scanziani et al., 2017).

In research areas where natural compounds must be preserved, various chemical flushing methods are used prior to fixing the sample (Boës & Fagel, 2005). For soils of high fines content, this method is not ideal due to potential of washing out fines and disturbing the natural microstructure. The sensitivity of the sample, in terms of its potential for disturbance, is especially high in peat soils that are very loose and can be disturbed very easily. Rock samples and densely packed soils are more rigid; however, pore connectivity may be a difficult factor when considering resin impregnation. Silts present a challenge for void penetration due to the mix of sand-like grains and clay-like grains. Sand particles can range from rounded to angular, while clay particles may be plate-like. The large variation of shape and size and well-graded nature of the overall soil causes a natural infilling of voids. With pore sizes on the micro or even nano-scale, small changes in pore fluid chemistry and mechanical properties could destroy the natural fabric of the soil. It is imperative for the viscosity of the impregnated material to not be too high so as not to disrupt the microstructure of the soil material. Highly viscous fluids could cause sample bloating and increase pore pressures, leading to altered microarchitecture. Therefore, resin with a viscosity as close to 1 centipoise (cP) is ideal, which is the unit of viscosity for water (Jang et al., 1999).

Resin is commonly used for impregnation into soil and rock specimens (Jang et al., 1999; Boës & Fagel, 2005; Yamamuro et al., 2008; Yang et al., 2008; Fonseca et al., 2012; Gyland et al., 2013; Kyle & Ketcham, 2015; Paniagua et al., 2015; Reyes et al., 2017; Schimmelmann et al., 2018; Frost et al., 2019). Acrylic resin LR White (Tippkötter & Ritz, 1996) and epoxy resins EPO-TEK, and Spurr are common manufactured resins used in soils research, particularly for coarse-grained soils. LR White is more commonly used in the biological field for preserving natural structures. Spurr resin has a viscosity around 100 cP and requires some heat for curing. It has been shown that temperature changes, particularly in heating above 60°C, can cause densification of the soil sample (Jang et al., 1999; Zhou & Ng, 2016). EPO-TEK resin can cure at room temperature and has a viscosity around 100 cP (Yamamuro et al., 2008). Jang et al. (1999) noted that
EPO-TEK resin curing is limited to only dry samples, thus saturated undisturbed samples cannot be preserved using this resin. Fonseca et al. (2012) performed EPO-TEK resin impregnation on dry triaxial Reigate sand specimens and noted minimal change in volumetric strain during impregnation and after curing. While this is meaningful evidence that resin impregnation poses minimal disturbance to the specimen, more work must be done on saturated, fine-grained material to confirm this.

2.6 Imaging Techniques for Geomaterials

Imaging techniques for the visualization of fabric and microstructure have developed extensively throughout the technological age. This section discusses two types of imaging techniques that are common in geomaterials research. Scanning electron microscopy has been used to achieve high-magnification images of two-dimensional planes. Applications of SEM imaging for Fraser River Delta silts are shown. Advantages and disadvantages of this method are presented. The latest three-dimensional imaging technology of X-ray micro-computed tomography is reviewed and its application to particulate materials is discussed.

2.6.1 Scanning Electron Microscopy (SEM)

SEM imaging is a common technique for obtaining high spatial resolution images of geological samples (Reed, 2005; Reyes et al. 2017; Markussen et al. 2019). Specimens can be imaged in loose, dry powdered form, or as a polished “hockey-puck” shaped specimen. Secondary Electron (SE) SEM imaging is typically used for dried samples, whereas pucks are often prepared for Backscattered Electron (BSE) imaging. Preparing preserved pucks for analysis involves resin impregnation, sectioning, polishing, and coating with a conductive material. Since the sample is fixed, it can be moved and manipulated without worry of disturbing the captured fabric and microstructure.

Dry powdered images allow the user to observe surface textures, aggregations, and three-dimensionality of particles. Verma (2019) presented an SE SEM image of non-plastic Fraser River silt (Figure 2.17). It can be seen here that shadows form among the particle spaces due to the light that is shone down during imaging. This shadow effect
makes it difficult to segment individual particles accurately, as the darkness from a shadow may be mistaken for a particle edge digitally. While this method yields valuable information about the particle surface texture, it does not allow for specimen fabric and microstructure evaluation.

Figure 2.17 Secondary electron SEM image of dry, powdered non-plastic Fraser River silt (Verma, 2019)

Various polished puck SEM work has been performed on silt-sized materials, which has been proven to show good contrast for grain size, orientation, and textural information. A notable advantage of SEM is its ability to reach very high resolutions. Markussen et al. (2019) performed scans at a magnification equivalent to 0.25 μm resolution. This allows visualization of smaller particles, as well as more clear grain surface textures and grain edges. Yamamuro et al. (2008) used SEM imaging to observe the influence of reconstitution on silty sand samples. At 43x magnification, particles less than 100 μm in diameter were distinguishable. Fawad et al. (2010) performed SEM imaging on manufactured silt-clay mixtures with the intention of observing microfabric and degree of alignment (DOA) for rose diagram construction. Crushed quartz silt ranged in particle sizes from 4 – 40 μm diameter. The clay composed of kaolinite and mica/illite ranged in 0.4 – 30 μm diameter. At 250x and 1000x magnification, quartz and mica/illite grains are distinguishable, however individual grains of kaolinite are not. Huang and Wang (2016)
imaged silty sand samples with nanoparticles for liquefaction susceptibility improvement. At 2500x magnification, sub-micron textures were visible, however the field of view (FOV) was very limited to an image about 50 μm x 40 μm.

In addition to a decreased FOV at higher resolutions, 2D SEM has several other disadvantages. In Section 2.6, the potential for sample disturbance during resin impregnation was noted. Figure 2.18 shows a BSE SEM puck image of Fraser River silt, prepared by A. Seidalinova during her graduate studies at UBC Civil Engineering Geotechnical Research Laboratory under the supervision of Prof. Dharma Wijewickreme (Wijewickreme, 2019).

![Figure 2.18 Polished puck backscattered electron SEM images of Fraser River silt by A. Seidalinova](image)

While the image on the left appears to capture grain boundaries well, it is evident in the image on the right that the impregnated resin caused disturbance in the pore structure and displaced material in several areas. As SEM requires a very smooth surface for imaging, the sample must be polished (Echlin, 2011). This polishing may result in false grain surface textures as well as loss of finer-grained material as dust. During imaging, high-energy electrons are emitted towards the sample, which increases temperatures and can sometimes result in vaporization of unstable elements (Goldstein et al., 2012). This potentially results in loss of sample volume. 2D SEM is also limited to a single plane of observation, which does not allow for observation of full grain shape, orientation, or pore connectivity. Single-plane observation can capture the general
arrangement of particles but fails to capture aspects related to geotechnical engineering such as stress transfer and void distribution, and its relation to hydraulic conductivity. To combat this issue, researchers have sliced samples at intervals to obtain several 2D images of the same sample, and then digitally “stitching” these images together to generate a pseudo-3D representation (Jang et al. 1999). This has proven successful in some cases, however multiple slicing and polishing steps introduces an unknown amount of uncertainty to how the slicing may have affected particle fabric, microstructure, and surface texture. Since the thin section may slice through individual particles, it is impossible to capture the true minimum and maximum diameter. Therefore, there is uncertainty in the dominant axis of the particle orientation.

2.6.2 X-ray Micro-Computed Tomography (micro-CT)

Advances in imaging technology, paralleled by computing power, have led to the development of non-destructive 3D X-ray micro-computed tomography (micro-CT). X-ray micro-CT and its application to soil science has been reviewed by many, including Ketcham and Carlson (2001), Taina et al. (2008), Cnudde and Boone (2013), Helliwell et al. (2013), and Bultreys et al. (2016). The image resolution and quality obtained from X-ray are mainly dependent upon the following 5 factors:

- Beam energy
- Attenuation coefficient of the object under investigation
- Exposure time
- Ray path distance
- X-ray detector

Attenuation is related to the density and chemical composition of the material and is defined as the ability of the material to absorb incoming X-ray energy (Ketcham & Carlson, 2001; Cnudde & Boone, 2013). At the initial invention of X-ray in 1895 by Wilhelm Conrad Roentgen, the technology was used for medical imaging, where the patient was stationary, and the X-ray source and detector could move freely. While the original X-ray only had the ability to reproduce 2D images, the origination of the computer allowed for the development of X-ray computed tomography. This concept
meshed together 2D X-ray “slices” to generate reconstructed 3D images using computer algorithms. The first X-ray CT scans were conducted by Hounsfield (1973), and the advent quickly became widely popular and adopted by both researchers as well as practitioners. By the 1980s, scientists had branched from the medical and biological fields, and were performing X-ray CT scans on inanimate specimens. Initially, X-ray energies and exposure times were limited due to the risk of radiation to humans and other living specimens, which restricted the ability to achieve higher-resolution scans. However, with advances in technology and the risk factor of radiation exposure removed, X-ray CT scanners have been developed that can produce images at the micro-scale, and more recently, the nano-scale. For this thesis, X-ray micro-CT technology was used, and thus the equipment will be exclusively referred to as such hereafter.

As previously mentioned, medical X-ray CT imaging is performed with a stationary subject and a mobile X-ray source and detector. Conversely, laboratory X-ray micro-CT imaging is conducted with a stationary X-ray source and detector, and the object under investigation is fixed on a rotating stage. A schematic of a laboratory X-ray micro-CT scanner setup is shown in Figure 2.19 below. By restricting movement of the source and detector, this allows the user greater control for more precise scanning at higher resolutions (Ketcham & Carlson, 2001; Cnudde & Boone, 2013; Helliwell et al. 2013).

![Figure 2.19 Schematic of a laboratory X-ray micro-CT scanner, showing the field of view of the sample (modified from Cnudde and Boone, 2013)](image-url)
There are three types of X-ray micro-CT scanners that operate with stationary sources and detectors that can be categorized into the following: laboratory without scintillator, laboratory with scintillator, and synchrotron. While synchrotron X-ray micro-CT can achieve very high, nano-scale resolution, it is still relatively new, and devices are more expensive and less prevalent. A laboratory X-ray micro-CT scanner without a scintillator utilizes an X-ray tube which generates a cone beam that travels a distance, passes through the object, and travels a distance, directly to the detector, as shown in Figure 2.19. This configuration relies solely on geometric magnification to generate an image. A laboratory X-ray micro-CT scanner with a scintillator uses the same cone beam, however in between the object and the detector there is a scintillator, which converts the attenuated X-rays into visible light. This light is then captured by the detector to generate an image. The scintillator allows for optical magnification, which increases the potential resolution. This is by far most common type of scanner used in soil science due to its capabilities for increased magnification. Some laboratory X-ray micro CT scanners and their producers include Xradia 520 Versa (ZEISS, Jena, Germany), XT H 225 (Nikon Metrology Inc., Brighton, MI, USA), SKYSCAN 1272 (Bruker, Billerica, MA, USA), phoenix nanotom m (General Electric – Research, Niskayuna, NY, USA), and Scanco µCT 100 (Scanco Medical, Brüttisellen, Switzerland).

Laboratory X-ray micro-CT scanners have proven advantageous for acquiring microscale visualization relatively quickly (5 – 40 hours) due to their target to detector size ratio. By having a physically small sample (1 – 10 mm diameter) and a large detector, attenuated beam intensities are captured and processed simultaneously across the detector. Due to this and the development of faster computer processing units (CPU), microscale imaging is becoming more common in the engineering arena.

2.6.2.1 X-ray Micro-CT for Particulate Materials

Computed tomographic imaging has been used widely in the medical and biology fields for observing anatomy of living specimens. It shows high contrast between bone and surrounding tissue, and due to the 3D nature of the image reconstruction, it allows the user to observe the anatomy in much higher detail and intricacy than simple 2D X-ray
images. As such, the application for X-ray micro-CT to particulate materials has been developed extensively since it provides a non-destructive view into the natural microarchitecture of the sample under analysis. Most importantly for silt-sized materials, the objective is to capture complete individual particles and their defined edges. This is dependent on the resolution of the scan, which is a function of the available energy as described in the previous section.

Flannery et al. (1987) demonstrated some of the first applications of X-ray micro-CT to 200 μm diameter glass beads as well as sandstone and coal samples. Some striations were visible in the coal sample imaged at 2.8 μm resolution. Since this pioneering study, X-ray micro-CT has been applied to other fields involving particulate materials such as agriculture, additive manufacturing, concrete development, and pulp and paper. Obtaining high resolution scans, while also achieving high quality particle edge detection, have been challenging for silt-sized materials (Nazhat & Airey, 2011). Table 2.2 outlines several research studies, their minimum grain size, and the achievable resolution.
Table 2.2 Review of application of X-ray micro-CT imaging of particulate materials

<table>
<thead>
<tr>
<th>Researchers</th>
<th>Material of Interest</th>
<th>Min. Particle/Pore Size (μm)</th>
<th>Resolution (μm)</th>
<th>Properties under Investigation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wang et al. (2003)</td>
<td>Asphalt concrete</td>
<td>-</td>
<td>300</td>
<td>• Damage parameters</td>
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<td>Iassonov et al. (2009)</td>
<td>Glass beads</td>
<td>2500</td>
<td>180</td>
<td>• Pore space features</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>• Scanning parameters</td>
</tr>
<tr>
<td>Kawaragi et al. (2009)</td>
<td>10% bentonite, 90% quartz sand</td>
<td>200</td>
<td>5.0</td>
<td>• Pore space</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>• Permeability</td>
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<td></td>
<td></td>
<td>• Micro-cracking</td>
</tr>
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<td>Luo et al. (2010)</td>
<td>Porosity of soil for land-use</td>
<td>750</td>
<td>234</td>
<td>• Macroporosity</td>
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<td>Peth et al. (2010)</td>
<td>Upper soil tillage layer</td>
<td>-</td>
<td>38.4</td>
<td>• Swelling, shrinking</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>• Pore space</td>
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<tr>
<td>Nazhat &amp; Airey (2011)</td>
<td>Sydney sand</td>
<td>60</td>
<td>5</td>
<td>• Void ratio</td>
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<td>• Compaction band</td>
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<td>Munkholm et al. (2012)</td>
<td>Silt loam porosity</td>
<td>&lt;10</td>
<td>60</td>
<td>• Porosity</td>
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<td></td>
<td>• Soil friability</td>
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<td>New Zealand pasture soils</td>
<td>50</td>
<td>48</td>
<td>• Macropore size distributions</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>• Pore shape</td>
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<td>Zacher et al. (2013)</td>
<td>Bentheimer sandstone</td>
<td>-</td>
<td>1</td>
<td>• Microporosity, permeability</td>
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<td></td>
<td>• Reservoir characterization</td>
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<td>Bock &amp; Jacobi (2013)</td>
<td>Aluminum open-cell metal foam</td>
<td>400</td>
<td>-</td>
<td>• Strut length</td>
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<td></td>
<td>• Pore shape</td>
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<td>Bossa et al. (2015)</td>
<td>Pore network of leached cement paste</td>
<td>&lt; 3.6</td>
<td>1.81</td>
<td>• Pore network heterogeneity</td>
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<td>• Connectivity</td>
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<td>• Channel size</td>
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<td>van Dalen &amp; Koster (2015)</td>
<td>Aerated emulsions for food products</td>
<td>200</td>
<td>8.0</td>
<td>• Bubble size over time</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>• Percent composition of air</td>
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<tr>
<td>al Mahbub &amp; Haque (2015)</td>
<td>Gippsland, Australia sand</td>
<td>120</td>
<td>14.28</td>
<td>• Microstructure evolution during compression</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>• Void ratio/particle size distribution</td>
</tr>
<tr>
<td>Authors</td>
<td>Sample/Additive</td>
<td>Vickers Hardness</td>
<td>Microhardness</td>
<td>Key Findings</td>
</tr>
<tr>
<td>-------------------------</td>
<td>---------------------------------------</td>
<td>------------------</td>
<td>---------------</td>
<td>------------------------------------------------------------------------------</td>
</tr>
</tbody>
</table>
| Fonseca et al. (2016)   | Reigate sand                          | 100              | 5             | • Force chain stress transmittance  
                             |                        |                  | • Fabric tensor                                                              |
| Zhang & Jivkov (2016)   | Hydrating cement paste                | 5.0              | 0.5           | • Pore size                                                                   |
| Reyes et al. (2017)     | Porphyry copper ore                   | -                | 8.9           | • Fracture network                                                            |
|                         |                                       |                  |               | • Elemental composition calibration between SEM and EDX                      |
| Bernier et al. (2018)   | Manufactured powdered metal additives | 26               | 0.7           | • Particle size                                                               |
|                         |                                       |                  |               | • Pore size                                                                   |
| Markussen et al. (2019) | Conglomerate and arenite rock          | -                | 4.9           | • Porosity network                                                            |
|                         |                                       |                  |               | • Cementation                                                                 |
|                         |                                       |                  |               | • Mineralogy                                                                  |
Table 2.2 highlights the wide range of applicability to pore space analysis and identifying particulate arrangements. In concrete and rock imaging, smaller particle grains and cementation agents such as bentonite makeup a portion of the sample, however these studies focused primarily on separating the solid phase from the pore space (Kawaragi et al. 2009; Bossa et al. 2015; Zhang & Jivkov, 2016). This is much easier to complete in terms of phase segmentation since the density of pore space and solid materials differ, for example Bock and Jacobi (2013) report change in density between air and aluminum as 0.00119 g/cm³ and 2.7 g/cm³, respectively. Validation of accurate solid/pore separation included comparing known void ratios of specimens, for which there was consistently good correlation. In rock and ore imaging, large variation in mineral specific gravity ($G_s$) allow for more accurate segmentation, such as bornite ($G_s = 5.06$) and quartz ($G_s = 2.65$), as presented by Reyes et al. (2017). This highlights the necessity for high energy, high contrast imaging parameters.

Successful particle segmentation of sand-sized sediments was shown by Iassonov et al. (2009), al Mahbub and Haque (2016), Fonseca et al. (2012, 2016). This was validated through comparing digital grain diameter to known material diameters, and matching grain size distribution curves from laboratory and digital segmentation methods. The resolution is the limiting factor for segmenting the smallest possible true particle volume. This further reinforces the requirement for an image resolution of 2 μm or less for silt-sized material, while still capturing a representative elemental volume of the overall specimen for fabric characterization.

2.7 Image Processing Software

Several image processing software have been developed internally and are commercially available for different applications. Some open-source codes for image processing include: 3DMA, VG Studio Max, Blob 3D, Quantim4, DTM, and ImageJ (Cnudde & Boone, 2013; Kyle & Ketcham, 2015; Houston et al. 2017). ImageJ is commonly used for 2D particle assessment, and its 3D counterpart, Fiji is also available. Fiji does not include advanced image filtering options or segmentation modules that were required for this research. Dragonfly (Object Research Systems, 2019) is a new
software package that is available as a limited version for free for research purposes. It has open-source code editing capabilities which can be advantageous for more personalized manipulation of datasets. As it is very new, tutorials and validation studies using this software are limited. In addition to this, the free version does not contain advanced image filtering options. Some software are also limited to either porosity or particle analysis.

Considering the above, Avizo 9.7 by Thermo Fischer Scientific (TFS) was the chosen software for conducting image processing, particle segmentation, and quantitative analysis. Avizo has been used by several geomaterial researchers including Luo et al. (2010), Fonseca (2011), Nazhat and Airey (2011), Zacher et al. (2013), al Mahbub and Haque (2016), Bultreys et al. (2016), Shah et al. (2016), Reyes et al. (2017), Scanziani et al. (2017), and Markussen (2019). Avizo was noted by Houston et al. (2013) to be one of the only software packages that yielded both particle size distribution and pore volume fraction data. Even if Avizo is not exclusively used for all image processing steps, researchers will often use it to apply particular filtering algorithms. It includes the most comprehensive filtering modules required for this research application, as well as many interactive features that optimize analysis for the user. Such features include Image Stack Processing, Recipe Creation, and the Segmentation editor. Other packages include the XPoreNetworkModeling extension as well as the applied geology TFS software PerGeos for rock analysis. Due to its extensive user base, Avizo, and medical-counterpart Amira, has an extensive tutorial database, engineering help desk, and practice datasets that were essential for the application of image-based processing for this research. The methodology used in Avizo 9.7 is covered in detail in Section 4.0.

2.8 Overview

The above chapter has reviewed key concepts of monotonic and cyclic shear response of soils, highlighting that fabric and microstructure changes due to reconstitution have an evident effect on silt behaviour in particular. It has identified that sample preservation and 3D imaging is the next frontier for understanding and attempting to quantify fabric and microstructure. There have been limitations in silt research due to its smaller
particle size and unique behaviour. Due to advancements in technology and computing power, the opportunity is now there for non-destructive three-dimensional imaging and segmentation of individual silt particles. The following chapters will cover the methodology and results associated with this research project.
3.0 Material Tested and Laboratory Procedures

This chapter focuses on the steps taken to obtain field samples and conduct laboratory procedures to prepare Fraser River Delta silt specimens for analysis using X-ray micro-CT. Section 3.1 describes the geological history of the material tested, including the depositional and morphological environment. A review of how the soils were obtained through the drilling and CPT programs is presented. Soil characterization and mineralogical X-ray diffraction data are also reported. Section 3.2 discusses sample preparation for both undisturbed and reconstituted specimens and presents the overall testing program. Section 3.3 introduces the sub-sampling methods that were attempted for containing an appropriate sample for the X-ray micro-CT device. Section 3.4 presents the resin impregnation procedure that was used for preserving specimen fabric and microstructure. This chapter is concluded with an overview of all the samples that were prepared and imaged during this research project.

3.1 Source Material

Fraser River Delta silt, particularly in the Richmond, Surrey, and Langley areas, has been a soil of interest due to its shown macroscopic behavioural differences as a result of fabric and microstructural changes. Figure 3.1 shows a map of locations where geotechnical field investigations have been undertaken to obtain samples of soil – work undertaken over the last 15 years at UBC as a part of a comprehensive research program to study the mechanical response silts (Wijewickreme et al. 2019). Silts retrieved from Site 2 (identified using yellow circle, Figure 3.1) were used in the testing for the present research program; it should be noted that Site A (identified using blue square, Figure 3.1) is the same sampling location as Site 2. This site is located in the south of Surrey, British Columbia, near the intersection of 40 Ave and 168 St.
3.1.1 Geological History

The Lower Mainland region of British Columbia where the site is located was subjected to high overburden pressures from glaciers of the last glaciation that occurred 11,000 years ago during the Pleistocene epoch (Mathews et al. 1970; Clague et al. 1983; Williams & Roberts, 1988). Since then, these over-consolidated glacial materials have been overlain by the relatively recent (< 11,000 years) Holocene sediments of the Fraser River delta and alluvial system. Holocene soil stratigraphy of the delta in this region can reach depths of up to 305 m (Clague, 1996). The surface materials consist of either overburden fill materials in urbanized areas or peat or bog sediments in farming areas. The underlying material is dependent on the region of interest and is a result of several depositional processes (Boggs Jr., 2006; Nichols, 2009). Areas near the riverbanks consist of depositions of mainly sands, sandy silt mixtures, and gravels in some places. In regions further away from the river channels, floodplain materials such as overbank silts, with some interbedded clays, are encountered. Closer to the outlet of the Fraser, Serpentine, and Nicomekl Rivers into the Strait of Georgia, tidal flats of

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**Figure 3.1** Map of Lower Mainland, British Columbia, and site locations for source material in this research study and previous studies

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**Legend**

- Sanin (2010)
- Soysa (2015)
- Verma (2019)
marsh and silt deposits are noted (Clague et al. 1983; Williams & Roberts, 1988). At
greater depths, interbedded sands, silts, and clayey silts are encountered. As this
research is concerned with the upper 10 m of the soil stratigraphy below the ground
surface, all soils under investigation can generally be considered to be normally
consolidated or slightly overconsolidated. This state of consolidation was shown to be
applicable for the silts obtained from the subject site through a series of 1-D
consolidation tests performed by Soysa (2015) and Verma (2019).

3.1.2 Geotechnical Field Investigations and Undisturbed Soil Sampling

Subject site (Site 2) is located within the flood plains of the Nicomekl and Serpentine
Rivers, both of which meander roughly from the northeast to the southwest and outlet
into Boundary Bay north of White Rock. Geotechnical field investigations conducted at
the site in 2013 comprised of electrical seismic cone penetration testing with pore water
pressure measurements (SCPTu), and conventional mud-rotary drilling with fixed piston
sampling using thin-walled, sharpened-edge, stainless steel tubes (76.2 cm in length,
with an inner diameter of 7.32 cm) having no inside clearance. Details of this testing
programs can be referred to in Soysa (2015) and Verma (2019). The cone penetration
tip resistance ($Q_t$), friction sleeve stress ($F_s$), friction ratio ($R_i$), and porewater pressure
($U$) were gathered and plotted with respect to depth are plotted in Figure 3.2. As shown
in Figure 3.2, the water table is just below 1.0 m depth. Initial $B_q$ Soil Behaviour Type
(SBT) classification by Robertson (1990) identified 1.0 – 15.9 m as mostly sensitive fine-
grained material, with some small layers of silt or sand mixtures. According to the
updated SBT classification system by Roberston (2016), depths from 1.0 – 12.0 m
contain silt and clayey silt (Soysa, 2015; Verma, 2019). This investigation led to a
targeted soil sample retrieval program within the silts identified in the depth range from
2.6 – 3.2 m (Tube #S2) and from 3.4 – 4.0 m (Tube #S3), as shown by the highlighted
zone in Figure 3.2. In order to further classify the soil type, laboratory soil
characterization including index testing was conducted, which is described in the next
section.
Figure 3.2 Cone penetration testing profile of Site 2/A (modified from Soysa, 2015 and Verma, 2019)

3.1.3 Grain Size Distribution and Index Testing

Extensive grain size distribution and index testing has been performed on Fraser River Delta silts in the UBC Graduate Geotechnical Laboratory by Sanin (2005), Soysa (2015), and Verma (2019). Previous work on these soils by these researchers also includes the following:

- Undrained triaxial testing
- 1-D consolidation testing
- Monotonic direct simple shear testing
- Cyclic direct simple shear testing

In order to eventually evaluate the feasibility of X-ray micro-CT to capture silts, it was important to gather past soil properties, index parameters, and grain size distributions of
Fraser River Delta silt samples. A hydrometer test was performed on material passing the No. 10 sieve from Tube #S2 (depth range 2.6 – 3.2 m) to confirm its grain size distribution with the previous studies. Figure 3.3 compares grain size distributions from previous research studies and the soil investigated for this thesis. It can be seen that silt from Tube #S2 conforms well with other studies, confirming consistent material type.

Percent fines is considered as any material less than 2.0 μm in size. In accordance with data gathered on Site 2 (Site A), the assumed properties and index parameters for the soil used in this study are presented in Table 3.1. Moisture content was taken during extraction of undisturbed specimens, which will be discussed in Section 3.2.1. No additional Atterberg limit testing was performed on specific samples.

Figure 3.3 Collection of grain size distributions as per previous studies on Fraser River Delta silts
Table 3.1 Soil properties and index parameters of soil used in this study

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Site 2/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Depth (m)</td>
<td>2.6 – 4.0</td>
</tr>
<tr>
<td>In-situ water content, WC (%)</td>
<td>40 – 44</td>
</tr>
<tr>
<td>Specific gravity, Gs</td>
<td>2.75</td>
</tr>
<tr>
<td>Plasticity index, PI</td>
<td>7</td>
</tr>
<tr>
<td>Percent fines (%)</td>
<td>15</td>
</tr>
<tr>
<td>Unified soil classification</td>
<td>ML</td>
</tr>
<tr>
<td>In-situ overburden stress, σ_v’ (kPa)</td>
<td>30 – 42</td>
</tr>
<tr>
<td>Normally or over consolidated</td>
<td>NC</td>
</tr>
</tbody>
</table>

3.1.4 X-ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis was performed on silt samples from Tube #S2. XRD is a commonly used technique for quantifying the mineralogical composition of samples. For the study of fabric and microstructure, it can be useful to identify the mineralogical make up of the soil under investigation for a number of reasons. Some research has been performed to correlate XRD analysis with other imaging techniques such as SEM and X-ray micro-CT. This method has proven valuable for cross-correlating various mineral densities, as shown by Kyle and Ketcham (2015), Reyes et al. (2017) and Markussen et al. (2019). Due to varying hardness and chemical composition, minerals have a characteristic crystalline structure. It would be beneficial to see if particle arrangement is predictable based on the percent distribution of mineral types.

XRD quantitative phase analysis was performed at the UBC Department of Earth, Ocean, and Atmospheric Sciences on dry silt passing the No. 40 sieve. The results are presented in Table 3.2, along with their specific gravity (Gs) values as per Klein and Philpotts (2013).
Table 3.2 X-ray diffraction quantitative mineralogical phase analysis results

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Composition (%)</th>
<th>G_s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>35.6</td>
<td>2.65</td>
</tr>
<tr>
<td>Plagioclase (Albite; Andesine)</td>
<td>27.9</td>
<td>2.62</td>
</tr>
<tr>
<td>Illite-Muscovite 2M1</td>
<td>11.6</td>
<td>2.8</td>
</tr>
<tr>
<td>Clinohlore</td>
<td>6.0</td>
<td>2.81</td>
</tr>
<tr>
<td>K-Feldspar (Orthoclase)</td>
<td>5.6</td>
<td>2.55</td>
</tr>
<tr>
<td>Augite</td>
<td>3.7</td>
<td>3.25</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>2.6</td>
<td>2.6</td>
</tr>
<tr>
<td>Actinolite</td>
<td>2.1</td>
<td>3.1</td>
</tr>
<tr>
<td>Dolomite – Ankerite</td>
<td>1.3</td>
<td>2.85</td>
</tr>
<tr>
<td>Andradite</td>
<td>1.0</td>
<td>3.9</td>
</tr>
<tr>
<td>Gypsum</td>
<td>0.8</td>
<td>2.32</td>
</tr>
<tr>
<td>Talc*</td>
<td>0.8</td>
<td>2.75</td>
</tr>
<tr>
<td>Pyrite*</td>
<td>0.6</td>
<td>5.0</td>
</tr>
<tr>
<td>Lizardite*</td>
<td>0.4</td>
<td>2.55</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100.0</strong></td>
<td></td>
</tr>
</tbody>
</table>

*Indicated minerals are present in such small concentrations that the certainty of the XRD analysis is low

The bulk of the silt material is composed of quartz, plagioclase feldspar, and illite-muscovite, for a total of 75.1% of the sample. The top 5 percentage of minerals also have a very similar G_s, averaging to a value of 2.69. Illite, muscovite, and kaolinite, which are all clay-type minerals, comprised 14.2% of this sample. It should be noted that only a very small specimen, around 10.0 g, was used for this analysis, therefore there can be a bias based on where the specimen was sampled from in the dry, crushed material. The potential for identifying different minerals in X-ray micro-CT analysis based on their G_s, hardness, cleavage, and natural crystal shape, is further discussed in Section 5.10.

3.2 Specimen Preparation

This section outlines the steps developed for preparing laboratory specimens that were to be sub-sampled for X-ray micro-CT imaging. It discusses the steps taken for extracting undisturbed specimens, as well as preparing reconstituted slurry deposited samples. A new cylindrical specimen mold was designed for 1-D consolidation, and its dimensions are discussed.
3.2.1 Preparation of Undisturbed Specimens

Undisturbed specimens were extracted from the field tube samples carefully using an upright extruder which uses a hydraulic piston. The piston is placed at the bottom of the thin-walled stainless-steel tube sampler, and the undisturbed specimen is carefully pushed out the top end. From visual inspection, the initial portion that was extruded was cracked and disturbed (Figure 3.4), thus this portion was removed carefully using a wire saw and used for reconstituted specimen preparation. Approximately 3.5 cm was then extruded and placed aside for further trimming (Figure 3.5).

The sample was then trimmed further using a soil lathe and the standard sharpened-edge testing ring 7.0 cm in diameter was pressed down into the extracted portion. Excess soil around the top, bottom, and sides was trimmed, and the moisture contents were recorded. This process was undertaken following a typical specimen preparation process that would be performed for behavioural testing using the DSS apparatus. These steps were kept as consistent as possible in order for this technology to translate to typical laboratory procedures.
Figure 3.4 Disturbed portion of specimen extracted via hydraulic piston from thin-walled sharpened-edge sampling tube

Figure 3.5 Undisturbed sample prior to further trimming by stainless-steel ring and soil lathe
3.2.2 Method of Slurry Deposition for Preparation of Reconstituted Specimens

Specimens were prepared according to the slurry deposition method as initially proposed by Kuerbis and Vaid (1988) for sands and adapted by other geotechnical researchers at UBC including Sanin (2005, 2010), Soysa (2015), and Verma (2019).

Fraser River Delta silt was first dried and carefully crushed by mortar and pestle. As material was available in limited quantity, approximately 300.0 g of material was weighed each time and mixed with approximately 250.0 g of water. The soil was mixed with enough water to ensure that the entire sample would remain saturated during the preparation phase, resulting in a thick, soupy texture. The specimen was kept under vacuum for approximately 24 hours to remove any air bubbles and occasionally stirred, after which it was removed from vacuum and allowed to settle under its own weight for another 24 hours. During this time, a thin film of water would develop at the top of the specimen, and this excess water was removed carefully with a small pipette prior to spooning the sample into its cylindrical consolidation mold.

3.2.3 Specimen Mold to Generate Reconstituted Silt Sub-Specimens for X-ray Micro-CT Imaging

The focus was placed on extracting a representative sample sufficient for X-ray micro-CT scanning purposes. Through laboratory testing experience, about the top and bottom 0.25 cm of the post-consolidation specimen height may be disturbed while removing the top cap and porous stone, as well as removing the specimen from the base plate. Such disturbance can cause smearing and shearing of the sample at that interface, altering the fabric and microstructure.

Typical standard 1-D consolidation and UBC-DSS devices use a specimen 2.0 cm in height (Figure 3.5). It was important to consider that the slurry could consolidate in the order of 25% upon loading to vertical effective stresses in the order of 200 kPa. If excluding 0.5 cm in height for top and bottom disturbance, only 1.0 cm of specimen at the center would be usable from a 2-cm height specimen for fabric and microstructure imaging. Therefore, it was judged that a taller specimen holder to result in a post-
consolidation height of at least 5.0 cm would be preferable (i.e., to ensure that at least a
4-cm undisturbed height would be available to extract sub-specimens for 3D analysis).

After paying due attention to the above considerations, the dimensions were fabricated
according to a pre-existing base plate and porous stone with drainage valve, which
included a sealing ring. The specimen height is 11.4 cm which is 1.5 times the inner
diameter of 7.6 cm. These dimensions were well in accordance with the available
loading frame.

3.2.4 One-Dimensional Consolidation

A stress-controlled oedometer loading frame was used for standard 1-D consolidation
testing of the reconstituted specimens according to ASTM D2345/D2345M-11 (2011).
Typical stress-controlled consolidation requires an almost instantaneous increase in
load; however, since the loading system herein was pneumatically controlled, the
regulator was manually turned as quickly and smoothly as possible to the target load
with minimum time elapsed for the process. The applied vertical force on the specimen
was measured using a load cell and the resulting vertical displacement was measured
using a linear variable displacement transducer (LVDT). The applied vertical load on
the specimen and associated vertical displacement registered in the transducers were
acquired digitally using a computer-interfaced data acquisition (DAQ) system. The
loading frame and DAQ are shown in Figure 3.6.
The water level of the specimen was maintained to match with the upper porous stone throughout the consolidation period in order to ensure fully saturated conditions. The main purpose of this 1-D consolidation process was to simply generate a consolidated silt specimen, ultimately with the intention of obtaining sub-specimens for imaging at various stress states and observing the changes in fabric and microstructure. Since the consolidation characteristics of Fraser River Delta silt have been extensively studied by other researchers at UBC (Sanin, 2010; Soysa, 2015), no specific effort was placed to obtain time versus settlement data from the consolidation tests. Once the desired load was reached, the specimen was removed from the consolidation frame and extracted from the cylinder. At this point, the final height was recorded, and the specimen was ready for sub-sampling. During sub-sampling, moisture content would be taken.

3.3 Specimen Sub-Sampling for X-ray micro-CT

X-ray micro-CT scanning requirements limited the size of sample able to be imaged. In order to optimize the image resolution as will be discussed further in Chapter 4.0, a
maximum sample diameter of 5.0 mm was required. As such, sub-samples had to be taken from larger laboratory specimens as described in the previous section. This section covers the several types of sub-sampling tubes considered, as well as tube dimensions that were investigated. Similar to how sampling disturbance must be minimized in the field when extracting undisturbed specimens, the sub-sampling method was also required to minimize disturbance in order to capture the fabric and microstructure of the original sample. The method of sealing the sub-specimen samples to maintain moisture content during travel is also discussed.

3.3.1 Sub-Sampling Tube Type

As indicated earlier, the sub-sampling method has to minimize disturbance during sampling, and moreover, the tube-material type had to be acceptable for use in the X-ray micro-CT scanner. A high-density contrast between tube and inner material would be ideal in order to accurately distinguish between the two mediums. However, if the outer tubing material is very dense, such as the stainless-steel used for in-situ sampling, the X-ray source energy will be highly absorbed, thus affecting the quality of imaging of the inner material. With this in mind, metal containers were not considered. It was most ideal to have a sub-sampling tube of lower density to the inner material to minimize X-ray absorption. Simultaneously, from a geometric point of view, the end of the tube had to be sharp enough to penetrate the soil and the rigidity of the material had to be quite high to ensure that the silt sample could not be squished or disturbed. No commercially available/manufactured acrylic or plastic tubing were available to meet the specifications required. Considering the aforementioned requirements, the following three material types were investigated:

- Thin-plastic drinking straws
- Custom glass tubing
- Custom 3D printed tubing

The thin-plastic drinking straws were purchased from a local grocery store. The custom glass tubing was cut and sharpened at the UBC Department of Chemistry Glassblowing Services. The 3D printed tubes were designed on SOLIDWORKS (Dassault Systems,
2019) converted to .stl files, and printed at UBC Electrical and Computer Engineering 3D printing services. Drinking straws are commonly made of polypropylene, which is a thermoplastic polymer with a density around 0.90 g/cm$^3$. Typical borosilicate glass used for chemistry glassware has a density of 2.23 g/cm$^3$. The 3D printing material is polylactic acid (PLA) filament which has a density of 1.24 g/cm$^3$. The drinking straw and 3D printed PLA achieved good imaging contrast with the inner silt specimen, while the glass material is much closer in specific gravity to some minerals within the silt specimen. In addition to this similar density, due to its reflectivity, glass can cause some disturbance with the X-ray source, and is therefore not an ideal material for X-ray micro-CT scanning.

### 3.3.1.1 Sub-Sampling Tube Material, Diameter, and Wall Thickness

Various iterations of specimen diameters and wall thicknesses for each material were attempted for sub-sampling and are shown in Table 3.3. The different types of sub-sampling tubes are shown below in Figure 3.7. The wall thickness of the straw was too thin to determine via digital caliper therefore it was confirmed by cutting through 10 straws, stacking them, measuring this reasonable cumulative thickness, and dividing the value by 10.

<table>
<thead>
<tr>
<th>Tube type</th>
<th>B (mm)</th>
<th>T (mm)</th>
<th>B/t</th>
</tr>
</thead>
<tbody>
<tr>
<td>Drinking straw</td>
<td>5.0</td>
<td>0.135</td>
<td>37.04</td>
</tr>
<tr>
<td>Glass tube A</td>
<td>10.0</td>
<td>1.5</td>
<td>6.67</td>
</tr>
<tr>
<td>Glass tube B</td>
<td>8.0</td>
<td>1.0</td>
<td>8.00</td>
</tr>
<tr>
<td>3D printed A</td>
<td>4.0</td>
<td>0.2</td>
<td>20.00</td>
</tr>
<tr>
<td>3D printed B</td>
<td>4.0</td>
<td>0.5</td>
<td>8.00</td>
</tr>
</tbody>
</table>
It can be seen in Figure 3.7v that the 3D printed tube A cracked towards the tip that was designed to be sharpened. The ability to print the sharpened edge was limited by the resolution of the 3D printing nozzle, resulting in cracking. The other end of the tube, however, was sturdy enough to extract some soil specimen, as shown. This cracking issue could be avoided in the future by simply excluding the sharpened edge. Due to the limitation imposed by the printing nozzle, a thicker-walled tube was printed. 3D printed tube B was deemed unsuccessful, as there was zero soil recovery with a wall thickness of 0.5 mm. As previously discussed in Section 2.2.1 and in work shown by Baligh (1985), the vertical strains may have been too great at the interior, thus fully disturbing the soil as it was penetrated through the laboratory specimen. Glass tube A also had a very low B/t ratio (6.67) however it was easily able to penetrate and recover soil specimens. Perhaps it was in part due to the smooth nature of the inner wall surfaces of the glass tube, as well as the wider diameter. As mentioned above, glass was not a preferable material for X-ray micro-CT scanning, and since the inner
diameters of these tubes was greater than 5.0 mm, they were not used further in this research. Based on these considerations, the drinking straw showed to be a viable candidate for sub-sampling due to its excellent recovery and B/t ratio. The downside to this method is that the walls are not rigid, and the sample must be handled with great care during transportation and when preparing for scanning. Further research needs to be performed using a higher-resolution 3D printer to obtain a rigid and smooth container with walls of around 0.2 mm thickness.

3.3.2 Sealing Methods

Once the sub-sampled specimens were prepared, they had to be sealed at the ends to maintain moisture and hold the soil in place during transportation to the X-ray micro-CT scanning facility. Following typical procedures for sealing field tube samples, paraffin wax was used to seal the ends of the straws. However, the wax was heated to around 90°C, and moisture loss was noted in the form of evaporation inside the straws (Figure 3.8). As an alternative method, UHU brand “sticky tack”, which is commonly used for mounting posters, was used to seal the samples. In order to check the ability for either method to preserve moisture content, two samples were sealed and held in a “moist room”, which is maintained at high humidity (close to ~100% at a temperature of ~22°C) by automatic spraying of water. After 5 days, the moisture contents were compared to their initial specimens from which they were sampled from. These results are shown below in Table 3.4. A 14.56% difference in moisture content was noted as a loss from the wax-sealed samples compared to a 5.99% difference in tack-sealed samples. Therefore, it was judged that sticky tack could be used as a viable method for temporary sealing in the future. After sealing with either method, the sample would be wrapped in cling wrap, labeled, put in a Ziploc® bag, and placed in the moist room until it was sent out to the scanning facility (Figure 3.8). In addition to wax and sticky tack sealing, resin sealing and impregnation was also investigated, which is presented in the next section.
Table 3.4 Comparisons of sealing method for maintain moisture content

<table>
<thead>
<tr>
<th>Sample type</th>
<th>Moisture content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial specimen</td>
<td>32.39</td>
</tr>
<tr>
<td>Sticky tack sealed</td>
<td>30.36</td>
</tr>
<tr>
<td><strong>Difference</strong></td>
<td><strong>-5.99</strong></td>
</tr>
<tr>
<td>Initial specimen</td>
<td>39.71</td>
</tr>
<tr>
<td>Wax sealed</td>
<td>33.93</td>
</tr>
<tr>
<td><strong>Difference</strong></td>
<td><strong>-14.56</strong></td>
</tr>
</tbody>
</table>

Figure 3.8 Drinking straw samples sealed with wax and sticky tack; circle indicates some evidence of evaporation (left) and samples wrapped in cling wrap (right)

3.4 Resin Preparation and Sample Preservation Procedure

Resin impregnation is a common technique for preserving fabric and structure in higher, permeable, granular material, as well as fractured rocks, as mentioned in Section 2.5. This section focuses on a new type of resin that was attempted to be used for specimen impregnation and preservation. It outlines the key characteristics that were beneficial in this resin for its application to silts, and well as the laboratory procedure conducted. This section also discusses various trials and notable results arising thereof, as they pertain to the applicability of resin impregnation for preserving silts for X-ray micro-CT analysis.
3.4.1 Resin Characteristics

Sample preservation was an important initial task to be undertaken for this research for several reasons. Preservation of the sample would allow for scanning reproducibility if the sample were to be imaged at several facilities. If the specimen could be fully impregnated with resin, then the fabric and microstructure could be transported and imaged without disturbance. While sealing techniques such as waxing are widely used, moisture can still escape. It was intended that this method of preservation could be performed immediately after an undrained, saturated test was performed. Important factors to consider for the choice of resin were the following:

- Very low viscosity (close to 1 cP)
- Chemical inertness
- Minimal flushing cycles to reduce potential for loss of fines
- Ability to cure under room temperature
- Little to no expansion of the resin material during curing

Henkel Loctite IS 535 RTC resin (hereafter referred to simply as “the resin”) was selected for impregnation testing due to its adherence to the criteria outlined above and in Section 2.5. It is a hydrophobic liquid, meaning it repels water. Historically used in the car parts industry, this resin has been used as a sealant for micropores in cast metal parts to prevent leakage of contained fluids. It is a methacrylate monomer that cures at room temperature with the addition of an acceleration agent (“the accelerator”) under anaerobic conditions (Loctite, 2016). The accelerator, while visibly more viscous than the resin, did not affect the overall viscosity of the solution due to its very low concentration. Multiple flushing stages for specimen preparation are not required. Properties of interest found in the technical data sheet (Loctite, 2016) for the resin are presented in Table 3.5.
Table 3.5 Resin properties in uncured and cured states

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Uncured</strong></td>
<td></td>
</tr>
<tr>
<td>Density at 25°C, g/cm³</td>
<td>1.0</td>
</tr>
<tr>
<td>Surface tension, dynes/cm</td>
<td>36.9</td>
</tr>
<tr>
<td>Viscosity (cP)</td>
<td>5 – 15</td>
</tr>
<tr>
<td><strong>Cured</strong></td>
<td></td>
</tr>
<tr>
<td>Density at 25°C, g/cm³</td>
<td>1.1</td>
</tr>
<tr>
<td>Glass transition temperature, °C</td>
<td>38</td>
</tr>
<tr>
<td>Coefficient of thermal expansion, K⁻¹</td>
<td>100 – 140 x10⁶</td>
</tr>
<tr>
<td>Design limit continuous temperature, °C</td>
<td>177</td>
</tr>
</tbody>
</table>

The density of the resin in the uncured state at 25°C was almost equal to that of water, which was ideal. A viscosity range between 5 – 15 cP was a significant improvement to the resins previously presented in Section 2.5, which had viscosities around 100 – 300 cP. The surface tension of water is 72 dynes/cm, which is higher than that of the resin. A cured density of 1.1 g/cm³ meant that the resin would yield good contrast for soil material during X-ray micro-CT imaging. The glass transition temperature and design limit continuous temperature were not of high concern as the cured samples were always kept at room temperature. The coefficient of thermal expansion shows that the material is relatively soft and can show some expansion. However, due to the preliminary nature of this study, strain gauges were not utilized and changes in volume were noted to not have been significant as the sample was confined within a rigid glass tube.

As this was the first time this particular resin was being used for applications to soil science, the recommended laboratory procedures had to be modified to accommodate small silt samples while still preserving their fabric and microstructure. These procedures are discussed below.

### 3.4.2 Resin Procedure

The standard resin preparation and soil preservation procedure is outlined below and depicted in Figure 3.9.
Initial documentation provided by Henkel (personal communications with D. Smith, 2018) recommended the addition of a minimum of 0.024 g of accelerator per 1000 mL of resin volume. Due to the small scale of experimentation, at any time only 10 to 50 mL of resin was used.

Several tests were performed in order to calibrate the resin curing process. Due to the typically industrial scale of this particular resin impregnation, large vats and temperature-controlled vacuum chambers are used. According to technical specifications, the resin is meant to be cured in a water bath at approximately 54°C. Warmer water was in fact proven to accelerate the curing process. However, the curing was attempted at room temperature (21°C) in order to prevent moisture loss from the soil samples. Initially, the curing tests were performed in a Terra Universal Dual Purge System anaerobic chamber (Figure 3.10, left). This method required a large amount of nitrogen due to its size, and resin curing took over 3 hours in some cases. A small jar with a nitrogen inlet and purge outlet was designed to concentrate the nitrogen towards the resin (Figure 3.10, right). No pumping was required as the naturally heavier nitrogen would push out the oxygen from the container. All testing was performed under a fume hood in a controlled laboratory environment.
In the interest of time, it was considered preferable to have the curing occur within a 3-hour window. Testing was performed with a series of test tubes containing wooden stir sticks and roughly 3 mL of the mixed resin and accelerator (“the solution”). The test tubes were introduced into the anaerobic chamber at regular intervals. The wooden sticks were lifted every 5 minutes in order to check curing progress. The beginning of curing was deemed successful once the test tube was lifted along with the wooden stick (Figure 3.11i). It was important to limit the amount of times the solution was disturbed in order to prevent turbulence in the fluid state. Through several iterations, it was found that 5 times the recommended accelerator (0.0012 g accelerator per 10 mL resin) was needed in order to nearly fully cure 3 mL of solution in 90 minutes.

Once this ratio was established, the ability of the solution to cure with soil was tested in the miniature nitrogen chamber (Figure 3.11ii). Dry portions of Fraser River Delta silt were immersed in solution and air bubbles were visible escaping from the soil, indicating that the solution was entering some pore spaces. Initial tests showed the resin cured mostly around the soil/glass test tube interface as shown in Figure 3.11iii/iv.
Figure 3.11 (i) Cured resin in test tube, showing successful resin/accelerator ratio (ii) Soil samples curing in nitrogen chamber (iii) Cured resin around soil/tube wall interface (iv) Another view of small amount of soil impregnation with resin

Due to certain limitations of X-ray micro-CT scanning as discussed in Section 3.3.1, glass tubing was unable to be imaged; therefore, a technique had to be developed to remove the resin impregnated samples prior to scanning. The cured resin did not adhere to the test tube glass walls; thus, the samples were easily removed by breaking the tubing.
Several techniques were attempted to fully impregnate the soil specimens after it was noted that the resin would cure only near the walls. This was likely due to the resin finding the path of least resistance, meaning a head differential, or hydraulic gradient would be required to allow the resin to flow through the soil sample pore space. The various iterations of resin impregnation that were attempted are presented in the following section.

3.4.3 Observations, Results, and Lesson Learned

It was attempted to extend the idea of the conventional falling head test setup (ASTM D5084-16a, 2016) to create a hydraulic gradient across the soil specimen and allow for natural flow of the resin through the specimen (Figure 3.12). As the resin was hydrophobic, it was thought that it would displace existing water in soil pores well out of the pore space. Resin impregnation was visible at the top 1.0 cm of a 3.0 cm soil specimen. However, after 3 hours, the resin began to cure, which caused the top portion to solidify, restricting flow through the rest of the sample. For this type of procedure to be effective in the future, it became clear that the amount of accelerator should be reduced to allow more time for resin flow. Vacuum suction through the bottom of the specimen to encourage fluid flow was also attempted. Due to the slippery nature of the glass, the soil specimen would displace, even at very low levels of vacuum. A porous stone to prevent fines loss could not be manufactured small enough to accommodate a specimen of 1.0 cm diameter or less.

Some resin impregnation was visible at the surface for about the first 1.0 cm from the inflow end. Figure 3.13 shows 4X optical micrographs of the suspected resin and moist soil boundary, as well as some infilling of larger voids at the tube wall. In the left figure, the moist soil is on the left of the boundary as indicated, and the resin impregnated soil is on the right. One of these samples was imaged in the X-ray micro-CT, and its results are discussed in Chapter 5.0.
Figure 3.12 Falling head test for resin flow through sample (left) and portion of cured sample (right)

Figure 3.13 Optical micrographs at 4X showing moist soil/resin impregnated boundary (left) and resin infilling voids in soil specimen (right)
Since the resin begins curing in an anaerobic environment, it is possible that simply due to the lack of oxygen towards the center core of the sample, the resin began to cure prematurely rather than according to the controlled accelerator ratio and nitrogen purge. Additionally, it is possible that the lower surface tension of the resin did not allow it to fully penetrate in micro- and nano-scale pore spaces.

The soil and resin interaction did show some unique characteristics that differed from those that existed in the case of when soil pores contained water. When a sub-sampled moist core extracted from the main laboratory specimen via glass tube was immersed in resin, the soil core kept its form and did not disintegrate or visually swell as it would in water. With this in mind, soil “mini-cores” were simply immersed in a test tube of resin and allowed to cure. This cure time was within the initially determined calibration window for curing. These sub-sampled specimens were successfully encased in resin, regardless of whether the resin fully penetrated into the pore space.

Four sample types and their X-ray micro-CT feasibility were attempted using the above method. They are described in Table 3.6 and their advantages and disadvantages are discussed. Figure 3.14 shows a schematic of each method.

Initial X-ray micro-CT scans showed that the soil core must be held perfectly vertical during curing. If the core was tilted off this axis, then the detector would lose its point of reference on the sample as it rotated 360°C on the stage. Due to this, metal "cages" were formed in an attempt to hold the sample upright. While this may have been successful, the high-density contrast of the malleable metal wire crossing the FOV of the soil specimen during the scan caused interference, and therefore this method was deemed unsuccessful. The final method, Type D, used machined plastic “donut” which had an outer diameter closest to the inner diameter of the tube, and an inner diameter close to 5.0 mm to hold the sub-sampled straw in place. Plastic donuts at each end of the straw held the sample well in place. This was the most reliable cured resin sample for X-ray micro-CT, and its imaging results are later discussed in Chapter 5.0.
Table 3.6 Comparison of various cured resin sample types

<table>
<thead>
<tr>
<th>Type</th>
<th>Sample description</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Soil specimen diameter similar to inner diameter of tube</td>
<td>• Sample upright</td>
<td>• When breaking out of tube, not enough cured resin at walls to keep sample intact</td>
</tr>
<tr>
<td>B</td>
<td>Soil specimen titled in large tube</td>
<td>• None</td>
<td>• Sample tilted off vertical axis, cannot be imaged</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• No resin where sample touches tube wall; exposed to air once broken out of test tube</td>
</tr>
<tr>
<td>C</td>
<td>Soil specimen held upright with metal cage</td>
<td>• Sample upright</td>
<td>• Requires lots of sample handling, could cause disturbance</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>• Metal casing X-ray attenuation too high</td>
</tr>
<tr>
<td>D</td>
<td>Soil specimen contained in straw, held upright by plastic “donuts”</td>
<td>• Sample upright</td>
<td>• None</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• No sample disturbance; straw taken directly from sub-sampling lab specimen</td>
<td>• Some sample breakage main occur where donuts touch tube wall</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Sufficient cured resin thickness between tube wall and specimen</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Successful in X-ray micro-CT</td>
<td></td>
</tr>
</tbody>
</table>

Figure 3.14 Cured resin sample types
3.5 Testing Program

Several systematic imaging studies were undertaken in order to gain better understanding of the feasibility for X-ray micro-CT to image Fraser River Delta silts. Table 3.7 summarizes the imaging program undertaken, showing the types of samples prepared, their consolidation level, as well as tube type and sealing method. The first image was simply a moist soil lump that was prepared at the imaging facility and scanned in a straw. Both undisturbed and slurry reconstituted specimens were prepared in order to compare their fabric and microstructure. Various tube types were tested to observe any influence on image quality due to density contrast of materials. Some samples prepared in cured resin, as discussed in the previous section, were imaged in order to see whether the resin had an effect on specimen fabric and microstructure. Reconstituted slurry samples were prepared to three 1-D consolidation stress states in order to observe changes in fabric and microstructure with increased loading. A calibration study to aid in digital particle segmentation was also conducted and is discussed further in Chapter 4.0.
### Table 3.7 Summary of testing program for X-ray micro-CT samples

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Soil preparation method</th>
<th>Stress level (kPa)</th>
<th>Tube type</th>
<th>Sealing method</th>
</tr>
</thead>
<tbody>
<tr>
<td>MON-FRlump</td>
<td>Random moist lump</td>
<td>-</td>
<td>Straw</td>
<td>-</td>
</tr>
<tr>
<td>UBCO-FR-SD-100-3D</td>
<td>Slurry deposition of Tube #S3 material</td>
<td>100</td>
<td>3D tube</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-SD-100-Str</td>
<td></td>
<td>100</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-UD-Res</td>
<td>Spoil ends of undisturbed Tube #S3</td>
<td>55 – 75</td>
<td>Resin</td>
<td>Resin</td>
</tr>
<tr>
<td>UBCO-FR-UD-RG</td>
<td></td>
<td>55 – 75</td>
<td>Glass</td>
<td>Resin</td>
</tr>
<tr>
<td>MON-FR-beads-A</td>
<td>Slurry deposition with Tube #S3 material</td>
<td>-</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>MON-FR-beads-B</td>
<td>sieved between No. 200-400 and 1.0 mm</td>
<td>-</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-beads-A</td>
<td>glass beads (calibration study)</td>
<td>-</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-beads-B</td>
<td></td>
<td>-</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-beads-StrRes</td>
<td></td>
<td>-</td>
<td>Straw/resin</td>
<td>Resin</td>
</tr>
<tr>
<td>MON-FR-UD-Str</td>
<td>Undisturbed samples from Tube #S2</td>
<td>30 – 42</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>MON-FR-UD-StrRes</td>
<td></td>
<td>30 – 42</td>
<td>Straw/resin</td>
<td>Resin</td>
</tr>
<tr>
<td>UBCO-FR-UD-Str</td>
<td></td>
<td>30 – 42</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-UD-StrRes</td>
<td></td>
<td>30 – 42</td>
<td>Straw/resin</td>
<td>Resin</td>
</tr>
<tr>
<td>UBCO-FR-50</td>
<td>Slurry deposition of Tube #S2 material</td>
<td>50</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-100</td>
<td></td>
<td>100</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>UBCO-FR-200</td>
<td></td>
<td>200</td>
<td>Straw</td>
<td>Wax</td>
</tr>
<tr>
<td>PPC-FR-200</td>
<td></td>
<td>200</td>
<td>Straw</td>
<td>Tack</td>
</tr>
</tbody>
</table>

### 3.6 Overview

This chapter covered the extent of the laboratory preparation and procedures required to generate samples that would be feasible for X-ray micro-CT scanning. The source material was described and characterized according to previous studies as well as standard laboratory tests. X-ray diffraction analysis provided insight on the mineralogical composition of the material under investigation. Methods of sample preparation included undisturbed and reconstituted slurry deposition. Reconstituted silt specimens were prepared to 50, 100, and 200 kPa in order to capture how the 3D fabric and microstructure evolves with increased loading. In order to capture a representative elemental volume for scanning, sub-samples had to be extracted from the laboratory prepared specimens. Different types of sub-sampling tubes and materials were explored, and a standard drinking straw was identified as the current best and quick option for retrieving sub-samples.
Sub-samples for imaging were sealed with both wax and sticky tack; sticky tack was identified to be the better option for preserving moisture content of a sample. Resin impregnation and sample preservation techniques were also explored. A new, very low viscosity resin was used and proved to be successful at achieving sealing the silt specimens. A variety of cured resin sample preservation techniques were explored. The testing program for X-ray micro-CT imaging was outlined.

The next chapter will describe the non-destructive imaging procedure as well as the image processing and particle segmentation steps necessary for eventual quantitative analysis of the prepared silt specimens.
4.0 **Non-Destructive Imaging and Image Processing**

As identified in Chapter 2.0, X-ray micro-CT scanning was considered as the next frontier necessary for non-destructive visualization of 3D fabric and microstructure of silts. An imaging program was thus conducted on the samples described in Table 3.9 of Chapter 3.0. Section 4.1 outlines the scanning facilities that were collaborated with for this research. It also summarizes essential components of X-ray micro-CT scanners, their output results, and terminology that is commonplace in digital imaging. Section 4.2 describes image processing techniques that are required prior to extracting data from the images, such as different filtering methods. Three filtering methods and their results are defined and compared in terms of their applicability to the datasets acquired during this research program. Section 4.3 briefly addresses the computer and subsequent computer power that is required to conduct the filtering tasks mentioned in Section 4.2. It outlines the adaptative analysis measures that were taken prior to obtaining a more powerful computer that was able to conduct the image processing steps to the fullest degree. Section 4.4 discusses the chosen segmentation techniques that were used for digitally distinguishing individual grains. This completes the background work necessary prior to conducting quantitative individual particle analysis.

4.1 **Imaging Equipment**

Three high-resolution laboratory X-ray CT machines were used for capturing non-destructive images of the soil specimens. The first was the ZEISS Xradia 520 Versa located at the Geomechanics Laboratory in the Department of Civil Engineering, Monash University, Australia. Samples were shipped via international mail to Monash and image data was shared via Google Drive. A second ZEISS Xradia 520 Versa that was used was located at the Pulp and Paper Center (PPC) at UBC Vancouver.

The third scanner was the Xradia MicroXCT 400 in the Composites Research Network – Okanagan Laboratory, located at the UBC Okanagan campus in Kelowna. Samples were sent through inter-campus mail and image data was shared via remote desktop sharing application AnyDesk.
The specifications of the Xradia 520 and the Xradia 400 are compared in Table 4.1. Some modifications can be made; however, these are the standard specifications as presented by ZEISS (2018) and Xradia (n.d.). The Xradia 520 can achieve a higher voltage, however, it is limited to smaller samples in terms of size. Since the soil samples were light, allowable weight restrictions were not an issue. The Xradia 400 has greater manipulation for source and detector distance, however greater travel distances may compromise for resolution, large capacity datasets, and poorer image quality. It should also be noted that degradation of the energy source and detector occurs with increased use, and regular maintenance is required. This bias for degradation was not considered when analyzing image quality, but it should be noted that this could have an effect.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Xradia 520</th>
<th>Xradia 400</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage range (kV)</td>
<td>30 – 160</td>
<td>40 – 150</td>
</tr>
<tr>
<td>Maximum power (W)</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Sample stage load capacity (kg)</td>
<td>25</td>
<td>15</td>
</tr>
<tr>
<td>Source travel (mm)</td>
<td>190</td>
<td>400</td>
</tr>
<tr>
<td>Detector travel (mm)</td>
<td>290</td>
<td>400</td>
</tr>
<tr>
<td>Sample size limit (mm)</td>
<td>300</td>
<td>100</td>
</tr>
<tr>
<td>Spatial resolution (μm)*</td>
<td>0.7</td>
<td>~1.0</td>
</tr>
<tr>
<td>Minimum achievable voxel (μm)*</td>
<td>0.07</td>
<td>0.3</td>
</tr>
</tbody>
</table>

* Achievable resolutions on the highest level of magnification, and therefore not necessarily admissible for this application

4.1.1 Scanner Input Control Parameters

Several iterations were required prior to arriving at the standard input control parameters. In an attempt to achieve a better resolution using the Xradia 400, a 10X magnification was used and the source position was 41 mm, achieving a resolution of 0.983 μm. Unfortunately, the image quality was poor, and it was noted that the 10X magnification may have required further recalibration. With the assistance of the scanning technicians at each facility, standard input parameters used for scanning, as well as the time required and resulting image resolution, are shown in Table 4.2. These were deemed to be the optimal configurations for each scanner considering the image resolution, FOV of the specimens, and laboratory time frame.
### Table 4.2 Standard imaging control parameters for both scanners

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Xradia 520</th>
<th>Xradia 400</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pixel size (μm)</td>
<td>3.38</td>
<td>3.2</td>
</tr>
<tr>
<td>Voltage (kV)</td>
<td>80.15</td>
<td>60</td>
</tr>
<tr>
<td>Power (W)</td>
<td>7.01</td>
<td>30</td>
</tr>
<tr>
<td>Current (μA)</td>
<td>87.49</td>
<td>500</td>
</tr>
<tr>
<td>Optical magnification</td>
<td>4X</td>
<td>4X</td>
</tr>
<tr>
<td>Source to object distance (mm)</td>
<td>37.01</td>
<td>285</td>
</tr>
<tr>
<td>Detector to object distance (mm)</td>
<td>37.01</td>
<td>17</td>
</tr>
<tr>
<td>Exposure time (s)</td>
<td>2.0</td>
<td>30</td>
</tr>
<tr>
<td>Total scan time (hr)</td>
<td>17.5</td>
<td>24</td>
</tr>
</tbody>
</table>

There are some notable differences in the scanning parameters used. The source to object distance is larger for the Xradia 400, resulting in a larger FOV and higher power and current needed to reach the object. In addition to this, the exposure time for each scanning interval was 2 seconds and 30 seconds for the Xradia 520 and the Xradia 400 respectively. Exposure time is the length of time that the source beam is directed at the object. While this increases the brightness of an image, it can introduce higher noise in the image, which will be discussed further in Section 4.2. A longer exposure time therefore increases the total scan time.

The difference in scanning parameters also resulted in different FOVs for the cylindrical 3D datasets. The FOVs for the Xradia 520 and Xradia 400 were:

- Xradia 520 Monash: 3.3 mm diameter by 3.3 mm height
- Xradia 520 PPC: 0.85 mm diameter by 0.85 mm height
- Xradia 400: 6.4 mm diameter by 6.4 mm height

The larger diameter for the Xradia 400 meant that the full sample, including tube walls, could be visualized. A greater height meant that a larger representative elemental volume of the sample would be captured. The Xradia 520 captured the inner core of the sample, which was still advantageous for analyzing fabric and microstructure not disturbed by the sampling tube wall. Greater heights and widths can be achieved by stitching several scans together, however this increases scanning time and file size.
significantly, thus this option was not explored. One scan at a resolution of 0.869 μm was achievable with the Xradia 520 PPC scanner, and its results are presented in Section 5.0.

4.1.2 Image Resolution and Greyscale

Section 2.6.2 in the literature review covered the general configuration of an X-ray micro-CT machine. The sample is imaged in the upright position, and the source beam is aimed at the side, perpendicular to the long axis. Images are taken at a set number of intervals for a full 360° of rotation. For example, the operator may set the number of intervals to 2,000 (i.e., 2,000 steps to complete a rotation of 360°). The higher this interval for a full 360° of rotation, the higher the resolution and the longer the scanning time. The detector and computer then stitch and reconstruct these projections together, and the resulting image can be viewed as slices in the XY, XZ, and YZ planes. The XY-plane is considered the horizontal reference, and the XZ- and YZ-planes are considered the vertical references, which are perpendicular to each other. A typical XY-plane slice is similar to what would be observed in a petrographic puck image, while in an XZ- or YX-plane slice, a cross-section of the entire height of the sample is observed.

When a 2D digital image for X-ray micro-CT is generated, its output is a formulation of “slices” of greyscale pixels. A pixel, in 2D, is a digital square of a size directly correlated to its resolution. If an image has a resolution of 1.0 mm, it means that each individual pixel is 1.0 mm x 1.0 mm in size. Therefore, a data set that is 10.0 mm x 10.0 mm would contain 100 pixels of 1.0 mm² area. For 3D datasets, this three-dimensional “volumetric” pixel is termed a “voxel”, which is an equivalently sized cube. An example of a complete data set of 1986 x 2026 x 1964 slices (XY x XZ x YZ) is shown in Figure 4.1. The thickness of each slice is one voxel, which is the resolution of the dataset. In this example, each slice is 3.2 μm.
Figure 4.1 Dataset of sample UBCO-FR-SD-100-Str showing visualization in all three planes

Each individual voxel stores a level of grey that was produced from the X-ray micro-CT scanning and post-imaging reconstruction process. This greyscale is dependent on the input control parameters used for imaging, as outlined in Table 4.2, as well as the X-ray attenuation. X-ray attenuation data, also known in the medical field as Hounsfield units (Ketcham & Carlson, 2001), are the output grey values that are a direct result of the sample density. Samples containing materials of a wide variety of densities will yield a detailed range of grey levels. Air, for example, has a very low density, and thus will show up as black. Very dense metallic ores, such as gold (Gs = 19.0, Kyle & Ketcham, 2015) will show up close to white in resulting images. This pattern of X-ray attenuation data allows for distinguishing between various materials and particles, which is a key goal of this research.
Another important aspect to consider is the ability of the imaging system and post-imaging reconstruction to capture variations in tone to an acceptably high degree. Densities of common soils constituents and minerals are very similar, for example 2.55 for K-feldspar and 2.65 for quartz (Klein & Philpotts, 2013). This notion of “tone” refers to the “bits” of an image. The amount of grey tones in an 8-bit image is 255, which is calculated as $2^{8} = 256$. The maximum value is 255 as the range begins at 0. As such, by simply doubling the bit capacity of an image from 8-bit to 16-bit, an image with 65,535 ($2^{16} - 1$) different grey tones can be achieved. This greatly increases the level of microscopic detail that the X-ray micro-CT images can capture. While the file size will also increase, it is advantageous to have 16-bit images for the purpose of distinguishing between soil grains of similar densities. For analysis, it should be noted that the Xradia 520 produced 16-bit images, while the Xradia 400 produced 8-bit images which limited the selection of greyscale.

In image processing, this greyscale tone capacity is commonly presented as an intensity range histogram (IRH), an example of which is shown in Figure 4.2. The histogram shows the frequency of voxels in the y-axis and their corresponding greyscale intensity on the x-axis. As can be seen, this is a 16-bit image, since the intensity range goes up to 65,535. It should be noted that not all images contain the maximum amount of grey tones in a particular bit image; this is just to say that a 16-bit file has the potential to contain up to 65,356 variations in grey tone. The large scale of the y-axis can give appreciation to the fact that this dataset contains over 1 billion voxels.
A significant amount of information about the sample that was imaged can be extracted from simply looking at the IRH. Some researchers (Kyle & Ketcham, 2015; Reyes et al. 2017; Markussen et al. 2019) have been able to cross-correlate mineral densities with specific greyscale values, so long as the X-ray micro-CT input control parameters were kept the same, and if it would be possible to analyze an identical sample in a variation of mediums such as under petrographic microscope, XRD, SEM, and X-ray micro-CT (as discussed in Section 3.1.4 on XRD analysis). This greatly increases the ability to obtain near-true particle segmentation as long as the image resolution is sufficient.

By observing Figure 4.2, it can be seen that there is a significant portion of voxels within the 5,000 – 15,000 greyscale range. Since it is known that the sample consists of much more soil than voids and the majority of the greyscale is not black, indicating air or water, it can be reasonably concluded that the bulk of the soil within this sample has a greyscale value greater than roughly 5,000. After 16,000, there is a dip in frequency, which can be correlated to a change in mineral type. Once again, there is a decrease at around 36,000, however as the scale is logarithmic, less than 1,000 voxels in each histogram bin shows that these mineralogies are much less representative of the soil sample overall.
Phase segmentation, or intensity range partitioning, is a common tool used to distinguish between different phases of a sample. By partitioning different greyscale regions, the user can observe and manipulate only that specific material in Avizo software. The following sections will outline the image processing methods that were attempted in order to achieve the ultimate goal of separating voids (air and water) from soil.

4.2 Image Processing – Filtering

Prior to particle segmentation, the image must undergo filtering in order to improve the inherent noise that is generated during the scanning phase. Noise can generate unwanted artifacts, cause image blurring, and can often be visually described as “salt and pepper noise”. Noise as a result of electronic interaction is referred to as Gaussian noise, which most commonly affects greyscale values of digital images (Boyat & Joshi, 2015). Since all X-ray micro-CT images are post-processed as greyscale images, Gaussian noise is the most likely contributor to noise in these images. As such, many filtering algorithms attempt to offset this noise with comparative normalized Gaussian distributions. Gaussian noise is the result of photon energy interactions during the X-ray scanning process and can be minimized by optimizing input control parameters such as voltage and power (Ketcham & Carlson, 2001; Buades et al. 2005; Helliwell et al. 2013). Further detailing about the mathematics behind these methods, is beyond the scope of this thesis; however, the concept of Gaussian noise is important to note when choosing appropriate filtering methods for X-ray micro-CT image processing.

The basic concept of filtering involves improving image quality through algorithms that compare the greyscale characteristics of specified voxel areas. This area is known as a “kernel” which is a window size specified by the user and can be synonymous with a symmetrical matrix. For example, if a kernel size is specified as 3x3x3, a cube of 3 voxels wide, 3 voxels high, and 3 voxels deep will be observed computationally in that single step of the algorithm. The central voxel will be assigned new characteristics as a result of the filtering method. This is shown schematically in Figure 4.3. There is a natural trade-off when selecting kernel size; the smaller the kernel, the higher the
computation time. However, if the kernel is too large, then the central voxel can be greatly changed due to larger potential for greyscale variation in a large kernel. Larger kernel sizes may also blur out details, which is not desirable.

Figure 4.3 Schematic of 3x3x3 kernel and central filtered voxel

If the level of noise is known through scanning calibration methods, it can easily be removed digitally. Many filtering algorithms have been created to remove this noise; however each filter has advantages and disadvantages when it comes to removing graininess while also preserving object edges. Particularly in soil samples where the densities of each grain are very similar, it is sometimes difficult to distinguish grain boundaries. These two characteristics, graininess and edge preservation, are important aspects of image filtering and particle segmentation. The graininess of an image may interact with the smaller particle sizes, and this may lead to mistakenly identified individual particles. The resulting filtered image must also preserve the boundaries of different particles and voids; thus, it is important to not have the filtered image to be too blurry.

With these two traits in mind, 3 different filters were applied to the images and their results were analysed to determine which one was the most appropriate application. Some filters only have 2D capabilities, therefore the focus was placed on 3D filtering applications. While 2D filters can be applied to 3D datasets, this does not allow for continuity among slices in X-ray micro-CT images. Multiple filters can be applied to images in succession; however this can increase digital uncertainty. In order to simplify the process, only one type of filter was chosen, however future works may consider
multiple filters. The methods of filter selection are described, and their results are presented in this section.

4.2.1 Symmetric Nearest Neighbor (SNN) Filter

The Symmetric Nearest Neighbor (SNN) filter compares pairs of voxels opposite from each other that surround the central pixel of a given kernel size. It then selects one voxel out of the pair that has greyscale values closest to the central voxel. The resulting filtered value is the mean of the selected voxels whose values were closest to the central voxel. A 2D schematic of this process is shown in Figure 4.4 for a 5x5 kernel. Pixels 5 and 21, as well as pixels 7 and 19 are compared to central pixel 13. The closest values to the central value of 4 are 4 and 7, thus the resulting pixel value is a mean of 5. The ability of this filter to be effective in removing noise while still preserving edges is highly dependent of the size of the kernel that is chosen and whether there are large fluctuations in grey values across the defined symmetric neighborhood. If there are outlier greyscale values that are not near the value of any voxel, then this value is not ever considered in the calculation.

![Figure 4.4 2D diagram of SNN filter application (adapted from Hall, 2007)](image)

4.2.2 3D Median Filter

The 3D median filter uses a defined neighborhood of 6, 18, or 26 voxels to conduct its iterative filtering algorithm. Based on the selected neighborhood, it will take the median voxel greyscale value for the central voxel. An example of this process for a 6-voxel
neighborhood is shown in Figure 4.5. The central voxel has a value of 12, which is much different to the surrounding voxels, and is thus converted to a median value of 5. This process is iterated multiple times until an appropriate level of filtering is achieved. This method preserves known greyscale values within the image, as it does not generate new values (Lamas-Rodriguez et al. 2015), such as would be the case if it were to take the mean. The 3D median filter can be advantageous at removing unwanted noise and artifact voxels from the image. However, it can sometimes lead to over-smoothing, which is detrimental to edge delineation.

![3D median filter methodology for a 6-voxel neighborhood](image)

4.2.3 Non-Local Means (NLM) Filter

The Non-Local Means (NLM) filter was developed by Buades et al. (2005) as a way of taking into consideration the image as a whole. The NLM filter utilizes the similarity of neighborhoods surrounding the central voxel as a way of determining what the new greyscale value should be. Depending on the similarity of the neighborhood value and how drastically a change in voxel greyscale occurs over a distance between the central voxel and local neighborhood, a weight is given to this neighborhood which determines how much influence it will have on the resultant central voxel value (Thermo Fisher Scientific (TFS), 2018). A simplified schematic of this procedure is depicted in Figure 4.6. The central voxel and the grey values of its local neighborhood of radius = 3 pixels are shown in the middle in green. Examples of local neighborhoods A, B, and C are within the user-defined search window of radius = 18 pixels. The mean of the central neighborhood is 4.44, and the mean of local neighborhoods A, B, and C are 4.33, 7.11,
and 20.44, respectively. The NLM algorithm would recognize that neighborhood B is likely indicating a change in medium from the central neighborhood due to its large difference in grey values. These grey values would not have a high weighting assigned to them for influence on the central voxel. However, by recognizing this sudden change in greyscale, the NLM algorithm can detect that this is likely an edge.

Figure 4.6 Simplified schematic of the Non-Local Means filter

4.3 Computing Power

It was quickly realized during the initial data analysis phase that a powerful computer would be necessary for performing tasks such as visualization, processing, segmentation, and analysis. When considering the size of files and graphics required to process 3D datasets, the four most essential components to be optimized are the following, according to the Thermo Scientific Avizo Software 9 User’s Guide (TFS, 2018):

- Graphics processing unit (GPU), or the graphics card
- Central processing unit (CPU)
- Random access memory (RAM)
- Hard drive
Older graphics cards, such as the NVIDIA NVS 315 included in the older computer, simply do not have the capacity or compatibility for performing basic Avizo 9.7 tasks. Numerous error messages cited that the graphics card was unable to visualize and perform particular modules. The Volume Rendering module, for example, would cause the program to stall and crash on the older graphics card. TFS (2018) recommends a minimum of 4GB GPU memory, and thus the newly purchased Lenovo computer system included an NVIDIA Quadro P5000 graphics card with 16GB GPU memory. A Compute Unified Device Architecture (CUDA) device was required in addition to a powerful graphics card (Gastal & Oliveira, 2012). CUDA is integrated into NVIDIA graphics cards and is used to optimize computation in terms of time and memory. It is important to note that this device does not diminish the quality of the output image and is only used for accelerating specific algorithms. This thesis will not further expand on this topic; however, it was necessary to note the presence of this device for a later section regarding image processing.

TFS recommends the available RAM be a minimum of 6 to 8 times the dataset size for data computational storage. Communications with an Avizo representative (Lancon, 2018) revealed up to 10 times the dataset size is ideal to take all other processing tasks into consideration. The initial computer operated on only 32 GB of available RAM, which was insufficient for processing data sets larger than 3 GB. The memory capacity during computation would often be surpassed and the processing step would have to be aborted. The new computer contains 128 GB of usable RAM, increasing the file memory capacity 4-fold.

Finally, an efficient hard drive is important for accessing and saving large volumes of data. A standard SATA3 7200RPM hard drive was deemed sufficient.

### 4.3.1 Benefits of Increased Computing Power

Due to the limited memory and graphics card of the old computer, a less computationally efficient method of the Non-Local Means filter had to be used. This Non-Local Means filtering would take up to 14 hours to process an image of 980 horizontal slices, 1.9 GB in size. Occasionally, the system would crash, and the
execution would have to be aborted. Therefore, images were initially processed in batches of 100 slices, which would reduce the file size significantly and allow for reasonable computational time. However, with this smaller batch, 3D information of grains was not as complete or continuous, and the representative sample size was further decreased. While output information was limited, this method was still successful in showing the usability of the Avizo program for Fraser River Delta silt imaging.

Given newfound computational ability with the upgraded computer, image processing could be performed in greater detail and on larger data sets. Non-Local Means filtering on complete file sizes up to 4 GB was computed in under 1 hour. This allowed for full representation of the 3D volume. Previously, information for about 2,000 to 7,000 individual grains was rendered, while the new computer in some cases generated over 150,000 grains. This increase in output data demonstrated that a more robust data analysis tool would be required, thus the analysis in Excel (Microsoft, 2019) was switched to MATLAB (MathWorks, 2019).

### 4.4 Image Processing – Particle Segmentation

One of the main objectives for the imaging of Fraser River Delta silts was to extract individual grain data for quantitative analysis. Since the image is simply made up of digital voxels, a particle segmentation analysis must be conducted to train the program to recognize grain boundaries and voids. After filtering, interactive thresholding must be performed to distinguish between voids and solids. This methodology is described in this section. Furthermore, the watershed segmentation algorithm to fully segment particles is presented. The quantitative individual particle values that were extracted after each segmentation are also listed.

#### 4.4.1 Interactive Thresholding

The first step in particle segmentation is selecting the appropriate greyscale values that separate particles from voids. This creates a binary image where the voids are black, a value of 0, and the selected greyscale voxels are given a value of 1. All subsequent functions used to conduct the segmentation require this input, and only the values of 1
are evaluated. As Section 4.1.2 revealed, the greyscale can range up to 65,535, thus a systematic method must be developed for selecting the appropriate thresholding value.

If the sample type and imaging parameters are kept consistent, this threshold greyscale value can be determined in detail and used across all datasets. As mentioned previously, researchers have been able to cross-correlate greyscale threshold values with specific minerals (Markussen et al. 2019), maintaining a very consistent particle segmentation process. However, while developing an appropriate testing program for Fraser River Delta silts, a number of parameters varied. Namely, samples were imaged in two different scanners, one which yielded 8-bit images, and the other 16-bit. As mentioned in Section 3.3.1, samples were contained in tubes of various materials. The X-ray attenuation of these materials could have influenced the tone and quality of imaging. Finally, the composition of the acrylic resin may have had a slight effect on greyscale values. Due to the above, a different thresholding value had to be selected for each dataset.

The IRH is often used for distinguishing this threshold value. The choice of filter can greatly change the shape and values of the IRH, therefore it is important to analyze it before and after filtering. Iassonov et al. (2009) and Fonseca (2011) show that a valley between peaks on an IRH is indicative of a change in medium. The lower this valley, the easier it is to distinguish between different phases. Filtering will ideally further distinguish changes in medium, which will increase this dip in the histogram. A slice of sample UBCO-FR-UD-StrRes is shown in Figure 4.7, followed by the dataset IRH prior to and after filtering (Figure 4.8). As this is an 8-bit image, its range can go up to a maximum of 255. It can be noted from Figure 4.7 that the FOV of the scan included the resin surrounding the sample, which is likely the contributor of the spike in grey values around 18 of the IRH. A dip in frequency is further enhanced by filtering and is reduced from 8.0x10^7 to about 5x10^7 between the unfiltered and NLM filtered results, respectively. The dip occurs at around 29 in the unfiltered dataset, however is decreased to 26 in the NLM filtered histogram. This is therefore a good option to begin for interactive thresholding. This methodology, of user interpretation to determine the greyscale value in conjunction with observing the IRH, was used for all datasets.
Figure 4.7 Slice from sample UBCO-FR-UD-StrRes

Figure 4.8 Intensity range histogram of UBCO-FR-UD-StrRes before (left) and after (right) Non-Local Means filter
4.4.2 Watershed Segmentation

Once data binarization has been completed, the image processing can proceed to particle segmentation. Some challenges are presented when performing this computation. If the sample is very densely packed, then it becomes increasingly more difficult to distinguish these grain boundaries, as particles are in contact with each other over larger surface areas. Additionally, if the mineralogy is very similar, then the greyscale separation can be challenging. A commonly used tool is known as the watershed segmentation algorithm (Beucher & Lantuejoul, 1979). The theory of watershed segmentation simulates water being poured over a landscape with peaks and valleys. This is analogous to the structural arrangement of the binarized data from the interactive thresholding step. If a figurative digital catchment basin collects a large amount of water, then this is indicative that this region is a strong region containing 0 for void space or 1 for solid space.

The description of each processing step in the watershed segmentation analysis is beyond the scope of this thesis, however the steps taken, and input parameters for Avizo are summarized in Table 4.3. The steps were followed in accordance with the tutorial described by TFS (2018). Various parameters such as those required for the NLM filter were optimized according to user-interpretation. A sample workflow showing the results from each step is shown in Figure 4.9 for the middle XY slice of sample MON-FR-UD-Str.
Table 4.3 Watershed segmentation steps and program inputs

<table>
<thead>
<tr>
<th>Step</th>
<th>Segmentation Function</th>
<th>Parameter</th>
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<td>Interpretation</td>
<td>3D</td>
</tr>
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<td></td>
<td>Measures</td>
<td>User-specified</td>
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</table>
Figure 4.9 Full watershed particle segmentation analysis
4.4.3 Quantitative Label Analysis

Once the particles were segmented via the watershed algorithm, quantitative values for each particle could be extracted. The Label Analysis tool allows the user to pick from a wide range of pre-determined measures or create customs calculations. The values that were extracted for each particle are listed below in Table 4.4. These values were used for the data analysis in Chapter 5.0 to analyze grain size distributions, rose diagrams, and particle shape characteristics.

Table 4.4 Primary quantitative particle parameters extracted from Avizo

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
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</thead>
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<tr>
<td>Length3D</td>
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</tr>
<tr>
<td>Thickness3D</td>
<td>μm</td>
</tr>
<tr>
<td>Width3D</td>
<td>μm</td>
</tr>
<tr>
<td>LengthOrientPhi</td>
<td>°</td>
</tr>
<tr>
<td>LengthOrientTheta</td>
<td>°</td>
</tr>
<tr>
<td>Area3D</td>
<td>μm²</td>
</tr>
<tr>
<td>Volume3D</td>
<td>μm³</td>
</tr>
<tr>
<td>EqDiameter</td>
<td>μm</td>
</tr>
</tbody>
</table>

The length, thickness, and width measurements are the maximum, intermediate, and minimum 3D Feret diameters, respectively. The Feret measurement is the distance between two parallel tangent lines along the particle perimeter. The minimum Feret diameter is most comparable to grain size distribution analysis (Altuhafi et al. 2013). Thickness is the measurement orthogonal to longest axis (length). LengthOrientPhi and LengthOrientTheta are the angles of deviation of the length from the z- and x-axes, respectively (Figure 4.10). With both angles, it is possible to visualize which quadrant the particle vector is in. Assuming particle symmetry in both directions of the long axis, the phi angle ranges from 0° – 90°. Theta ranges from -180° – +180°. The area is the 3D surface area of the full particle. The volume is essentially the count of voxels that comprise a single particle, multiplied by the voxel resolution. The equivalent diameter quantitatively is the diameter the particle would be if it were a sphere, based on its determined 3D volume. The purpose for this parameter is discussed later.
4.5 **Filter Selection and Scanning Calibration**

A systematic approach was used to analyse the three digital image filters and their ability to reduce image noise and preserve edges. This method was cross-checked with the resulting IRH of each filtering process. This section presents the filter selection process and describes the scanning calibration procedure that was undertaken to ensure that the chosen filtering and particle segmentation analysis yielded accurate results of particle shape and volume.

4.5.1 **Initial Filter Application**

The SNN, Median, and NLM filters, as described in Section 4.2, were applied to a central slice of sample UBCO-FR-UD-StrRes. A zoomed in portion of the resulting filtered images are shown in Figure 4.11, compared to the raw slice to visually demonstrate their applicability. The SNN filter preserves the particle edges well, however does not remove much noise. The median filter over-filters for noise, blurring edges significantly. It is evident that the NLM filter strikes a good balance between reducing noise and maintain particle edges.
Figure 4.11 Application of three filter types to determine best option

The resulting IRH of each filter are also shown in Figure 4.12. Both the SNN and Median filter results in a reduction the greyscale levels from the available 243 to 184 and 225, respectively. This reduction further limits the greyscale levels available for interactive thresholding. The Median filter enhances the distinction between two phases, as shown around an intensity level of 25, while the SNN valley remains similar to the raw image IRH. The Non-Local Means filter was shown to be the most successful filter for enhancing the phase contrast while maintaining the same intensity range as the raw image, and thus was the selected filter for all subsequent analyses.
Figure 4.12 Intensity range histogram before and after three filtering methods

4.5.2 Particle Segmentation Calibration – Glass Beads

To calibrate the X-ray micro-CT scanning setup, image processing methodology, and particle segmentation analysis, glass beads of a known diameter were introduced into a soil sample. Fraser River Delta silt from Tube #S3 was first wet sieved within the No. 200 and 400 sieves, resulting in a known grain size distribution between 37 to 75 μm. The soil was prepared as a slurry and spooned into a small plastic mold. At two height intervals, glass beads of 1.0 mm diameter were placed in the sample. The sample was lightly consolidated with free weights overnight, and then straw sub-samples were taken from the specimen (Figure 4.13). As the placement of beads and area of sub-sampling was random, two samples were sent to each scanning facility to ensure that at least one straw specimen would contain at least one glass bead.
The scan of sample MON-FR-beads-A successfully captured two full glass beads within the FOV. The dataset was cropped to maintain efficient computation time and to focus on segmenting the glass beads. The final segmented image post-Label Analysis is shown after performing filtering, thresholding, and watershed segmentation (Figure 4.14ii). The glass beads are distinguished, and their volume rendering also presented in Figure 4.14iii and iv. For reference, glass bead 1 is the bead on the left, as depicted in Figure 4.14i. Figure 4.14iii shows manual measurements of the glass bead diameters. It should be noted that this slice was not cut directly through the largest cross-section of each glass bead, therefore these are not maximum diameters. The length, thickness, width, 3D volume, and equivalent diameter of the particles as determined by the label analysis are presented in Table 4.5.

Table 4.5 Quantitative results for glass bead segmentation

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Glass bead 1</th>
<th>Glass bead 2</th>
</tr>
</thead>
<tbody>
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<td>Length, μm</td>
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<tr>
<td>Thickness, μm</td>
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<td>Width, μm</td>
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<td>Volume, mm³</td>
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<tr>
<td>Equivalent diameter, mm</td>
<td>0.955</td>
<td>0.950</td>
</tr>
</tbody>
</table>
Figure 4.14 (i) Cropped dataset showing glass beads (ii) Completed label analysis (iii) Distinguished glass beads and measured diameters (iv) Volume render of glass beads

The glass beads were of similar density to the surrounding silt material; thus, it was difficult to achieve perfect segmentation of the glass beads from the particles that were in contact with them. Some particles are still touching the glass beads as shown in the 3D render, however the smoothness of the beads is still captured in some areas (Figure 4.14iv). Nonetheless, the image processing methodology was very successful in capturing diameters of roughly 1.0 mm. Considering the volume of a 1.0 mm diameter
sphere is 0.524 mm$^3$, the 3D volumes captured only deviate between 12.9 to 14.3% for glass beads 1 and 2, respectively. It is possible that there is some inconsistency with manufacturing of glass beads perfectly 1.0 mm in diameter. While neither glass bead was perfectly spherical, equivalent diameters of 0.950 mm and above prove that this segmentation method is very successful in capturing representative shape, diameter, and volume of individual particles.

### 4.6 Overview

This chapter presented a detailed summary of the steps that were taken for X-ray micro-CT image post-processing. The scanning facilities and parameters of the scanners used were stated. Basic definitions associated with 3D image analysis were reviewed. Different filtering methods to reduce image noise were compared, and based on this, the Non-Local Means filter was chosen as the most effective filtering algorithm. Details of interactive thresholding to separate voids from solids, as well as the watershed segmentation algorithm were described as the main methodology for particle segmentation. The quantitative results that were extracted for each data analysis were listed. A scanning calibration was performed with glass beads, which showed that the above methodology accurately captured particle boundaries, volumes, and diameters. The following chapter, Chapter 5.0, presents the results obtained from each dataset, and demonstrates the ability for X-ray micro-CT to represent fabric and microstructure in silts.
5.0 Digital Image Analysis

Once the laboratory and image processing methodologies were established, various specimens were imaged to assess and demonstrate the applicability of X-ray micro-CT scanning to image Fraser River Delta silts. This chapter presents the quantitative and/or qualitative findings as a result of this imaging research. Section 5.1 summarizes the scanning details of successful specimen scans, as well as discusses some lessons learned from unsuccessful scans. Section 5.2 presents the digital grain size distributions that were generated as a result of particle segmentation and compares these results to laboratory and past research findings. Sections 5.3 and 5.4 address other quantitative measures for analyzing fabric, namely void ratio and rose diagrams of particle axis orientations. The subsequent sections qualitatively discuss features of note from the imaging program, including wall effects, resin impregnation, and differences between undisturbed and reconstituted specimens. Sections 5.9 and 5.10 discuss the potential for X-ray micro-CT imaging of silts for quantifying particle shape characteristics as well as recognizing mineralogical differences in specimens.

5.1 Scanning Details

Several specimens were scanned at the three facilities mentioned in Section 4.1. The name codes are according to where the specimen was scanned, what type of material, as well as the method of preparation and tube type. UBCO-FR-SD-100-Str, for example, was a Fraser River Delta silt slurry deposited specimen consolidated to 100 kPa, subsampled and contained within a straw, and imaged at the UBCO Xradia 400 scanning facility. Table 5.1 below outlines the samples that were imaged, as well as whether the scan was a success, as indicated by Y or N (yes or no). Of the successful scans, their image bits, voxel size, dataset size, greyscale threshold value, and number of particles analyzed are presented. Snapshots of datasets and quantitative results are shown throughout. The following subsection discusses some lessons learned from scans that were deemed unsuccessful.
Table 5.1 Completed scans and image processing characteristics

<table>
<thead>
<tr>
<th>Specimen name</th>
<th>Scan success</th>
<th>Image bits</th>
<th>Voxel Size (μm)</th>
<th>Dataset size (μm)</th>
<th>Threshold value</th>
<th>Number of particles</th>
</tr>
</thead>
<tbody>
<tr>
<td>MON-FRlump</td>
<td>Y</td>
<td>16</td>
<td>3.38</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>UBCO-FR-SD-100-3D</td>
<td>N</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>UBCO-FR-SD-100-Str</td>
<td>Y</td>
<td>8</td>
<td>3.2</td>
<td>6282 x 6480 x 6352</td>
<td>45</td>
<td>136,880</td>
</tr>
<tr>
<td>UBCO-FR-UD-Res</td>
<td>N</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>UBCO-FR-UD-RG</td>
<td>Y</td>
<td>8</td>
<td>3.16</td>
<td>6039 x 6399 x 6333</td>
<td>38</td>
<td>239,637</td>
</tr>
<tr>
<td>MON-FR-beads-A</td>
<td>Y</td>
<td>16</td>
<td>3.38</td>
<td>3323 x 3407 x 2366</td>
<td>6,000</td>
<td>-</td>
</tr>
<tr>
<td>MON-FR-beads-B</td>
<td>Y</td>
<td>16</td>
<td>3.38</td>
<td>3323 x 3407 x 3346</td>
<td>8,000</td>
<td>-</td>
</tr>
<tr>
<td>UBCO-FR-beads-A1</td>
<td>N</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>UBCO-FR-beads-A2</td>
<td>Y</td>
<td>8</td>
<td>3.2</td>
<td>6346 x 6480 x 6339</td>
<td>31</td>
<td>-</td>
</tr>
<tr>
<td>MON-FR-UD-Str</td>
<td>Y</td>
<td>16</td>
<td>3.38</td>
<td>3323 x 3407 x 3346</td>
<td>6,000</td>
<td>113,040</td>
</tr>
<tr>
<td>UBCO-FR-UD-Str</td>
<td>N</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>UBCO-FR-UD-StrRes</td>
<td>Y</td>
<td>8</td>
<td>3.2</td>
<td>6346 x 6480 x 6339</td>
<td>32</td>
<td>96,522</td>
</tr>
<tr>
<td>UBCO-FR-50</td>
<td>Y</td>
<td>8</td>
<td>3.2</td>
<td>6346 x 6480 x 6349</td>
<td>27</td>
<td>72,080</td>
</tr>
<tr>
<td>PPC-FR-200</td>
<td>Y</td>
<td>16</td>
<td>0.869</td>
<td>847 x 879 x 854</td>
<td>14,475</td>
<td>16,099</td>
</tr>
</tbody>
</table>

5.1.1 Influence of Scanning Specimen Holder on Image Quality

As mentioned in Section 3.4.3 on specimen preparation procedure, it was essential to maintain an upright vertical axis of the specimen in order to achieve high image quality; even less than a degree deviation from the vertical axis could affect the resulting image. Figure 5.1 show two holder types used during imaging. The holder shown in Figure 5.1ii uses a clamp system. With a flexible straw used to contain specimens, minor deviations from the verticality were possible. In Figure 5.2 of a vertical slice obtained from the specimen UBCO-FR-50, it is clear that the specimen is slightly tilted off its axis, as shown by the deviation of the straw wall from the vertical FOV. The image slices shown in Figure 5.3 show some other examples of blurry images and unsuccessful scans. This finding proved that for future X-ray micro-CT imaging, a sturdier base and holder should be used for these small, fragile specimens.
Figure 5.1 Two specimen holder types for X-ray micro-CT scanning

Figure 5.2 Sample image slice showing deviation from central vertical axis
5.2 Grain Size Distribution

In the laboratory, grain size distribution of soils is determined in two ways; sieve analysis for coarse-grained materials, and hydrometer analysis for fine-grained materials. Mechanical sieving utilizes a stack of wire mesh sieves decreasing in opening size, and systematically weighs the mass of the material retained on each sieve. Hydrometer testing is conducted on materials passing the No. 200 sieve (75 μm). It
involves preparing a solution of material, dispersing agent, and water in a standard cylinder, and allowing the suspended solids to settle while recording how the density of the solution changes over a 24-hour cycle. Hydrometer analysis requires corrections for particle specific gravity, temperature, and effective depth of the hydrometer tool – all while estimating the particle diameter under the assumption of a perfect sphere.

Both the laboratory methods determine the amount of material retained/passing each designated sieve size based on mass or density, therefore a correlation had to be used for the digitally constructed grain size distribution obtained from post-processing and particle segmentation. Since the grain size distribution is simply a cumulative distribution based on total mass, true 3D volume was considered equivalent to mass, assuming uniform specific gravity. The Width3D (μm) data (minimum Feret diameter, as defined in Section 4.4.3) was first sieved based on particle diameter, and then the Volume3D (μm$^3$) of each particle which coincided with each sieve size was summed. The cumulative volume for each sieve size was then divided by total volume retained over all sieves, obtaining a percent passing by mass equivalent volume (%).

The digital grain size distribution for 5 specimens is shown in Figure 5.4, comparing them to laboratory obtained hydrometer data for this research as well as from previous researchers of Fraser River Delta silt. Appendix A contains a detailed script of the coding used for this analysis.
5.2.1 Resolution Effects

An important characteristic for individual particle and overall fabric analysis is to determine how many voxels are representative of a full particle volume of interest (VOI). This value is limited by the smallest particle diameter in the specimen and the maximum achievable image resolution. Fonseca (2011) and Ní Bhreasail (2013) indicated 10 voxels as the minimum, and Taylor (2016) mentioned up to 15 voxels. Other researchers, such as Bossa et al. (2015) have used a minimum of 2 voxels to represent a VOI. The largest voxel size (lowest resolution) for the scans was 3.38 μm, which would result in a minimum representative particle diameter of 33.8 μm if using 10 voxels for the VOI. This would result in not capturing over 50% of grain size distribution. Due to these limitations, the resolution cut-off for individual particle representation was selected as 3 voxels. Therefore, any Label Analysis data for particles of a minimum Feret diameter 3 times the voxel size was not analyzed for particle volume or shape characteristics. This cut-off value is shown as a dashed “interpolated” line on the grain size distribution plots, indicating that particle sizes as determined digitally between the resolution and cut-off size may not truly represent full particle volumes. This cut-off
resulted in the loss of resolution for the finer fraction less than 20% percent in the PPC-FR-200 case, up to around 36% in the MON-UD-FR-Str case.

5.2.2 Comparison to Hydrometer Data

The data shown in Figure 5.4 conforms well with the laboratory determined grain size distribution as well as previous studies on Fraser River Delta silt. The highest resolution dataset (PPC-FR-200) appears to underestimate grain size, yielding a curve showing finer-grained material. Interestingly, this specimen slurry was one of the last reconstituted samples prepared for this study. Previous research on fine-grained material has shown that the fines content tends to decrease with reuse of reconstituted material, as the fines can be lost as dust during the preparation and drying phases. This decrease in grain size distribution can therefore likely be attributed to the digital segmentation methodology.

Overestimation of grain size occurred in the digital samples in which particle segmentation was difficult due to either image quality or wall effects. In these specimens, such as UBCO-FR-50 and UBCO-FR-SD-100-Str, particle segmentation often resulted in large, false digital aggregations of particles, which is reflected in their grain size distribution. An example of this is shown in Figure 5.5.

Figure 5.5 Example showing overestimation of grain size due to false digital aggregation
The most agreeable dataset was MON-FR-UD-Str, which was the exact material that, after being sub-sampled for undisturbed 3D imaging analysis, was dried, crushed, and used for the laboratory hydrometer analysis which is presented. It should be noted that there is inherent variability in where the specimen was sampled for each sub-specimen, therefore variability is expected. Nonetheless, the digital grain size distribution curves all fall within the silt range, following a similar trend to the laboratory and previous hydrometer test data.

5.3 Void Ratio

The ability to accurately capture laboratory and digital void ratio would be promising for confirming proper segmentation between solid and void space. Digital segmentation appears to underestimate the void ratio in almost all cases. For example, the laboratory void ratio for specimen UBCO-FR-50 was 0.838, while the digital void ratio was 0.411. As the pore space can be on the nano-scale, it is not possible for the segmentation methodology to capture these sizes at the current imaging resolution. The void volume can be mistaken for smaller particles and be included in the particle segmentation process.

The void ratio calculation showed to be highly dependent on the chosen interactive thresholding value. In a 16-bit image of the specimen MON-FRlump, varying the greyscale cut-off between 5500, 6000, and 6500 resulted in void ratios of 0.272, 0.469, and 0.819, respectively. Visually to the user, these thresholding values were nearly identical in terms of distinction between the solid and void phases. When cross-referencing the IRH, the valley between phases in the histogram was also lowest around these values. Further steps were attempted in order to select this value using software-provided algorithms, however as the scanning input parameters all varied, it was difficult to automate this selection process. The visual method, while cross-correlating with the IRH, proved to be successful for particle segmentation during the initial methodology development as discussed in Section 4.0.

It can be seen in some UBCO images, where the entire width of the straw was in the FOV, that the specimen had either dried or was compressed due to the thin-plastic walls
of the straw. This made it very difficult to computationally distinguish between the void space that was due to the movement around the walls rather than the true sample void volume. In addition to this, the greyscale of the straw was sometimes similar to that of some particles. Some steps were taken to apply a “mask” to the volume fraction calculation of void and solid volume, in order to remove the straw and space between the straw wall and the sample. It is due to the aforementioned reasons that the void ratio calculations for the UBCO samples may not be accurate at this time.

5.4 Preferential Particle Alignment and Rose Diagrams

Preliminary evaluations on primary long axis orientations were performed in order to form rose diagram plots (as per Section 2.1). These plots would be useful in showing particle realignment with respect to changes in stress state and method of preparation. The $\psi$ angles, while presented from $0^\circ$ to $90^\circ$ from Avizo software (Figure 5.6iv), were converted to $0^\circ$ to $180^\circ$ depending on which quadrant the particle’s long axis was in. Therefore, $\psi$ angles $0^\circ$ and $180^\circ$ represent the most horizontal position. A sample coding script for this analysis can be found in Appendix A.

As the number of full particles that were analyzed varied per dataset, their percentage of occurrence in each $\psi$ angle range was determined relative to their total grain counts. Table 5.2 shows preliminary findings of particle alignment based on specimens consolidated to varying vertical effective stress ($\sigma'_v$) states of 50, 100, and 200 kPa. Figure 5.6 shows the corresponding diagrams for these datasets, as well as a review of the angle measurements in Avizo.
Table 5.2 Primary axis orientation ($\psi^\circ$) frequency at various $\sigma'_v$ consolidation stress states

<table>
<thead>
<tr>
<th>$\psi^\circ$</th>
<th>Angle frequency at each stress state (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50 kPa</td>
</tr>
<tr>
<td>0 – 15</td>
<td>10.65</td>
</tr>
<tr>
<td>15 – 30</td>
<td>13.51</td>
</tr>
<tr>
<td>30 – 45</td>
<td>9.93</td>
</tr>
<tr>
<td>45 – 60</td>
<td>6.91</td>
</tr>
<tr>
<td>60 – 75</td>
<td>3.74</td>
</tr>
<tr>
<td>75 – 90</td>
<td>0.00</td>
</tr>
<tr>
<td>90 – 105</td>
<td>1.18</td>
</tr>
<tr>
<td>105 – 120</td>
<td>1.74</td>
</tr>
<tr>
<td>120 – 135</td>
<td>7.54</td>
</tr>
<tr>
<td>135 – 150</td>
<td>10.76</td>
</tr>
<tr>
<td>150 – 165</td>
<td>20.15</td>
</tr>
<tr>
<td>165 – 180</td>
<td>13.89</td>
</tr>
</tbody>
</table>

Figure 5.6 Rose diagrams depicting distribution of $\psi$ angles for reconstituted specimens at consolidation stress states (i) 50 kPa (ii) 100 kPa (iii) 200 kPa and (iv) initial Avizo coordinate system
Although this is preliminary data, it is of interest to note that the rose diagram appears to become more symmetric with increasing stress state. At $\sigma'_v = 50$ kPa, there is greater affinity for angles ranging from $135^\circ - 180^\circ$. The 200 kPa specimen shows nearly symmetrical frequency of particle alignment in the angle ranges of $0^\circ - 45^\circ$ and $135^\circ - 180^\circ$; this suggests that the results seem to correlate well with the assumption that particles are more likely to horizontally rearrange at higher stress states.

Despite these observations, it is be noted that much particle data is ignored when performing these analyses due to the resolution cut-off described in Section 5.2.1. Prior to removing this data, it was noted that particle sizes less than the cut-off were most likely to have angles between $75^\circ - 105^\circ$. It appears that the software auto-classifies single-voxel cubic particles with a primary axis angle close to $90^\circ$. As such, these frequency percentages are very low comparatively. This computational bias should be noted for future analyses in classifying particle alignment, and associated improvements to quantification methods.

### 5.5 Wall Effects

Effects of the specimen walls could not be seen in the images conducted at the Monash and PPC laboratories as the tube nor the affected zone within the soil specimen was captured in the FOV. In contrast, the FOV of the imaging performed at the UBCO laboratory captured the entire soil specimen including the straw. As may be seen from Figure 5.7, the images seem to have nicely captured the definite smearing along the wall edges – i.e., proving the particle rearrangement parallel to the tube walls. Based on visual measurements of specimen UBCO-FR-SD-100-Str, it appears that approximately $1/10^{th}$ of the specimen diameter inwards from the wall is affected around the specimen, meaning $1/5^{th}$ of the entire diameter across shows realignment effects due to the sampling tube.
These wall effects were also evident during particle segmentation. It was clear that the fabric seems to have compacted around the wall boundary constraint, as the particles were so close together that they often could not be segmented successfully. Figure 5.8 below shows this phenomenon of disrupted particle segmentation due to wall effects in specimen UBCO-FR-50. In addition to this, the particles nearest the walls appear brighter (more white) likely because these areas get the highest energy flux when being imaged in the X-ray micro-CT scanner. This energy is decreased towards the central core of the sample, resulting in the central particles appearing less bright, even though they may have the same specific gravities as those towards the outside. This phenomenon was also more evident in 8-bit images, as the range of greyscale could only reach a maximum of 255. This made it difficult to accurately segment the particles, therefore oftentimes the dataset was cropped to only consider the central core of the sample. This method was still effective, as the overall intention of this research was to capture the fabric generated due to undisturbed and reconstituted specimens, and not specifically outside factors. Nonetheless, these observations can be translated to field
sampling methods, showing how deep into the specimen diameter the sampling wall has an effect.

![Figure 5.8 Influence of wall effects on segmentation process, showing inaccurate thresholding "halo".](image)

5.6 Differences Between Undisturbed and Reconstituted Samples

For both Tubes #S2 and #S3, specimens obtained from undisturbed samples as well as those derived from reconstitution using slurry deposition were imaged.

In typical images obtained at the micro-scale, it can be seen that natural segregation and banding is more evident in the specimens obtained from undisturbed samples (Figure 5.9). This could be due to seasonal variations as well as tidal conditions (Clague et al. 1983; Monahan et al. 1993) that affect sediment deposition processes in real-life situations – e.g., coarser sediment is deposited at riverbanks during the spring melt times when the river velocity is high, and finer material is deposited at calmer river flow points in the year such as the peak of summer. These seasonal laminations are commonly visible in lake deposits at the more macro-scale, however it is interesting to observe this at such a micro-scale. This micro-scale observation further supports the potential for tidal influence on type of sediment deposition (Monahan et al. 1993). As expected, this banding is not seen in the slurry deposited samples; their arrangement appears to be more random and well-graded throughout the height of the sample (Figure 5.10ii). This could be a possible reason for why void ratios for undisturbed
specimens are typically higher than reconstituted specimens at the same stress state. Since the fine-grained material is confined to these natural varves, infilling of void space does not occur as readily as when the specimen is intermixed in a slurry and then consolidated. These varves were captured in the MON-FR-UD-Str specimen, albeit on a smaller scan as the FOV was half the height of the UBCO scans. Figure 5.9 shows banding of coarser-grained material, indicated by the red arrows.

Figure 5.9 Sample vertical slices of undisturbed specimens showing preferential banding/segregation

Figure 5.10 Sample vertical slices of (i) undisturbed specimen and (ii) reconstituted specimen. No natural varving noted in the reconstituted specimen
Large, low density particles, likely organic matter such as wood, are noted in undisturbed specimens, as well as reconstituted specimens that were prepared using material that was not oven dried or crushed prior to sample preparation. By the second round of reconstituted slurry preparation, the presence of organic matter was reduced significantly. Since the organic matter is likely partially degraded, its low density and fragility likely led to it being crushed or disintegrated during over drying. As such, reconstituted specimens often do not contain these organic particulates, which appear to lead to relatively large localized void zones in undisturbed specimens, as shown in specimen MON-FR-UD-Str (Figure 5.11). The shape and size of these cavities closely match that of wood particles in other specimens, sometimes being larger than 1.7 mm in length.

![Figure 5.11 Organic particulate matter in undisturbed specimen MON-FR-UD-Str](image)

5.7 Influence of Resin Encased Samples on Specimen Fabric

Specimens UBCO-FR-UD-StrRes and MON-FR-UD-Str were undisturbed specimens sampled directly next to each other using straws, as shown in Figure 5.12. Specimen UBCO-FR-UD-StrRes, was cured upright in resin and scanned. Due to it being protected by the resin on the outside, it was evident that the straw could not be
disturbed, and the soil remained in contact with the straw wall in most areas. In some areas, space along the straw wall was noted, which would end in a horizontal type “fissure”. These fissures may have been caused by resin flowing along the wall/soil interface. These macro-scale fractures appear in both undisturbed specimens that were cured in resin (UBCO-FR-UD-RG and UBCO-FR-UD-StrRes). However, it should be noted that they only seem to occur in the coarser-grained varved areas, as indicated in Figure 5.13. It is likely that the pore sizes within these coarser-grained particles were larger, allowing resin to flow through these spaces with more ease.

As discussed in the previous section, relatively large void zones appeared to be a result of organic matter or partially degraded organic matter. Some of these internal void spaces in specimen UBCO-FR-UD-StrRes therefore do not appear to be due to resin impregnation or sub-sampling effects, as they also exist throughout the MON-FR-UD-Str specimen which was not impregnated with resin. The resin material likely preferentially flowed towards one of these cavities in UBCO-FR-UD-StrRes, as indicated by the red arrow (Figure 5.13ii), making it larger, however organic matter remnants are clearly visible in this area.

While the resin may have caused macro-fractures and minor infilling of degrading organic matter void spaces, it still appears successful in showing the preferential banding consistently seen among all undisturbed specimens imaged. In some internal core areas of the specimen, the resin does not seem to affect overall fabric, and the sample is comparable to the specimen scanned without resin impregnation (Figure 5.14, MON-FR-UD-Str).
Figure 5.12 Figures showing sub-coring of undisturbed specimens

Figure 5.13 Resin-impregnated undisturbed specimen UBCO-FR-UD-StrRes in (i) horizontal and (ii) vertical section
5.8 Influence of Wax Sealing on Samples

As is typically done in the field to preserve moisture, ends of sampling tubes are often sealed with paraffin wax. Following this example, sub-sampled straw ends were filled with wax for the specimens prepared for calibration purposes with glass bead inclusions (see Section 4.5.2). The subsequent X-ray micro-CT images revealed significantly large, round “cavities” in the specimens (Figure 5.15i). By comparing the greyscale threshold of wax on the outside of the sample, and noticing some particle rearrangement around these cavities, they were determined to be filled with wax. Space between internal specimen and tube walls were also noted to extend towards these cavity areas, indicating that wax had flowed along the straw wall.

A reasonable conclusion for why the wax congregated at these specific areas was due to the glass bead inclusions. Another slice of the dataset shows that wherever glass beads are near the tube walls, wax cavities are formed (Figure 5.15ii). Since the thin-plastic straw was flexible, the glass beads encountered near edges of the specimen stretched the tube walls, creating a space for which the wax preferentially infilled. This
finding further corroborates the need for relatively stiff tubing for sampling, as well using sticky tack for temporary sealing rather than wax.

![Images](image1.png)

**Figure 5.15** Horizontal slices showing (i) large wax-filled cavities around the tube walls and (ii) cavities near glass bead inclusions

### 5.9 Particle Shape Characteristics

For observing and classifying particle shape characteristics, several of the extracted particle data was used. Long, intermediate, and short axis lengths were classified according to Zingg’s chart, and nearly all particles fell within the equant and roller shape index classification. An example of this for specimen UBCO-FR-SD-100-Str is shown in Figure 5.16.
Other parameters typically used to characterize particle shape are roundness and sphericity. As roundness is not a computational measure in Avizo, it was calculated according to the criteria proposed by Hayakawa and Oguchi (2005), which statistically shows an 80% fit with typical Krumbein’s roundness as presented in Section 2.1.1 and further proven by Cruz-Matias and Ayala (2013). Equation 5.1 below shows roundness as a function of the particle’s three primary axes, as well as the volume and surface area.

\[
R = \frac{V}{S(abc)^{\frac{1}{3}}}
\]  

(5.1)

Power (1953) classified roundness according to the grades listed in Table 5.3 below.

**Table 5.3 Roundness classification according to Power (1953)**

<table>
<thead>
<tr>
<th>Grade term</th>
<th>Class interval</th>
</tr>
</thead>
<tbody>
<tr>
<td>Very angular</td>
<td>0.12 – 0.17</td>
</tr>
<tr>
<td>Angular</td>
<td>0.17 – 0.25</td>
</tr>
<tr>
<td>Subangular</td>
<td>0.25 – 0.35</td>
</tr>
<tr>
<td>Subrounded</td>
<td>0.35 – 0.49</td>
</tr>
<tr>
<td>Rounded</td>
<td>0.49 – 0.70</td>
</tr>
<tr>
<td>Well rounded</td>
<td>0.70 – 1.00</td>
</tr>
</tbody>
</table>
All particles for each dataset fell within the “very angular” to “angular” classification. An example of UBCO-FR-SD-100-Str comparing roundness to sphericity is shown in Figure 5.17. Roundness calculations were limited due to small digital aggregations around particles that were a result of the particle segmentation process. This was shown in the example of glass bead segmentation in Section 4.5.2. A current limitation of the methodology is that it is unable to capture perfect particle boundaries, which can be due to poor contrast between grains of similar density and low resolution. The particle 3D surface area was also likely overestimated due to these small aggregations, thus reducing the calculated roundness.

Figure 5.17 Roundness versus sphericity, according to Power’s (1953) roundness classification

Despite quantitative particle shape limitations, the raw X-ray micro-CT data was analyzed by rendering a 3D reconstruction of the higher density particles. Figure 5.18 shows that X-ray micro-CT is very successful in capturing grain shape and texture, as shown by many rounded particles in the FOV. The X-ray micro-CT imaging technique is also successful in capturing silt-sized bladed particles, as indicated by the manual dimensions. As such, the methodology can be further developed to overcome quantitative classification issues.
5.10 Particle Shape, Greyscale Value, and Correlations to Mineralogy

The shape of a soil particle will be a product of many factors, including source rock mineralogy, chemical and physical weathering, and transport and depositional mechanisms. Key mineralogical characteristics to consider with respect to the above are mineral cleavage, hardness, and specific gravity. Cleavage shows the most natural form of how a crystal will break along specific planes (Klein & Philpotts, 2013). Quartz, for example has glass, conchoidal cleavage, while potassium feldspar (K-feldspar) cleavage is blocky at 90° and biotite and similar micaceous family minerals show flaky, sheet cleavage (Figure 5.19).

![Figure 5.18 Raw data of particles prior to digital segmentation](image)

(i) Quartz, (ii) K-feldspar (Klein & Philpotts, 2013), and (iii) Biotite (St. John, 2011)

Figure 5.19 Hand samples of (i) quartz, (ii) K-feldspar (Klein & Philpotts, 2013), and (iii) biotite (St. John, 2011)
Hardness, as defined by Mohs in 1812, is the abrasiveness of particles on each other. Talc has the lowest hardness of 1, quartz a moderately hardness of 7, and diamond with the highest hardness of 10. Specific gravity is important as it relates to density, which translates directly to the greyscale value of a particle in X-ray micro-CT imaging.

When taking all of the above into consideration, as well as the XRD results obtained in Section 3.1.4, it may be possible to predict mineralogical assembly and grain shape through X-ray micro-CT correlation. Table 5.4 shows the XRD analysis results, as well as mineralogical characteristics according to Klein and Philpotts (2013).

### Table 5.4 XRD analysis and corresponding mineral characteristics

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Composition (%)</th>
<th>Gs</th>
<th>Hardness</th>
<th>Cleavage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>35.6</td>
<td>2.65</td>
<td>7</td>
<td>conchoidal</td>
</tr>
<tr>
<td>Plagioclase (Albite; Andesine)</td>
<td>27.9</td>
<td>2.62</td>
<td>6</td>
<td>blocky</td>
</tr>
<tr>
<td>Illite-Muscovite 2M1</td>
<td>11.6</td>
<td>2.80</td>
<td>2 - 2.5</td>
<td>thin sheets, tabular</td>
</tr>
<tr>
<td>Clinohlore</td>
<td>6.0</td>
<td>2.81</td>
<td>2 - 2.5</td>
<td>micaceous</td>
</tr>
<tr>
<td>K-Feldspar (Orthoclase)</td>
<td>5.6</td>
<td>2.55</td>
<td>6</td>
<td>90, blocky</td>
</tr>
<tr>
<td>Augite</td>
<td>3.7</td>
<td>3.25</td>
<td>5 - 6</td>
<td>87/93</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>2.6</td>
<td>2.60</td>
<td>2</td>
<td>massive</td>
</tr>
<tr>
<td>Actinolite</td>
<td>2.1</td>
<td>3.10</td>
<td>5 - 6</td>
<td>fibrous</td>
</tr>
<tr>
<td>Dolomite – Ankerite</td>
<td>1.3</td>
<td>2.85</td>
<td>3.5 - 4</td>
<td>rhombohedral</td>
</tr>
<tr>
<td>Andradite</td>
<td>1.0</td>
<td>3.90</td>
<td>6.5 - 7.5</td>
<td>conchoidal, angular</td>
</tr>
<tr>
<td>Gypsum</td>
<td>0.8</td>
<td>2.32</td>
<td>2</td>
<td>massive, tabular</td>
</tr>
<tr>
<td>Talc*</td>
<td>0.8</td>
<td>2.75</td>
<td>1</td>
<td>massive</td>
</tr>
<tr>
<td>Pyrite*</td>
<td>0.6</td>
<td>5.00</td>
<td>6 - 6.5</td>
<td>brittle, conchoidal</td>
</tr>
<tr>
<td>Lizardite*</td>
<td>0.4</td>
<td>2.55</td>
<td>4</td>
<td>fibrous</td>
</tr>
</tbody>
</table>

*Indicated minerals are present in such small concentrations that the certainty of the XRD analysis is low

Actinolite (2.1% sample composition) is considered as an example for correlating compositional percentage, specific gravity, hardness, and cleavage. In hand sample, actinolite appears as long, fibrous strands, as shown in Figure 5.20, which would anticipate an aspect ratio of the particle to be 0.5 or less (half the width to length). It has a hardness between 5 – 6 and a specific gravity of 3.10. As it has a relatively average hardness, it can be expected to not completely disintegrate during transportation and keep some sort of its natural form. In addition to this, its higher that average density may have yielded some resistance to weathering and transport, and it would appear brighter relative to other minerals in X-ray micro-CT. Illite and muscovite also show similar tabular form and have slightly higher than average specific gravities. However, due to their low hardness (2 – 2.5), and since these minerals are typically associated as
fine-grained, clay-forming minerals, it is unlikely that full mineralogical structures are captured at these image resolutions. Figure 5.21 shows a vertical slice of sample PPC-FR-200 at 0.869 μm resolution indicating some possibilities for minerals visually fitting the actinolite criteria described above as well as relative sample composition. These minerals show fibrous cleavage, are brighter compared to surrounding particles, and are present in low compositional amounts. High variability for mineralogical composition is possible depending on where the sub-sample is obtained for XRD analysis, however these values are currently the most representative mineralogical analysis available for this type of application. This cross-correlation technique is promising, especially if scanning parameters are kept consistent among samples of the same material to further correlate image greyscale with mineral density.

Figure 5.20 Hand sample of actinolite mineral inclusions, from Klein and Philpotts (2013)
5.11 Overview

This chapter presented the results obtained from the X-ray micro-CT pilot research program. Some lessons learned with respect to image quality were discussed. The particle segmentation methodology was proven successful in capturing digital grain size distribution which matched well with laboratory-obtained data and past research of Fraser River Delta silts. Preliminary findings for quantitative results on void ratio, rose diagrams, and particle shape characteristics were presented, showing that methodology could require further development for silt fabric quantification. Wall effects due to sampling and the thin-plastic straw tubing were noted, showing that about 1/5th of the total specimen diameter nearest the tube walls showed smearing and particle rearrangement parallel to the walls. Paraffin wax treatment also showed some adverse effects on specimen fabric, especially with coarser-grained inclusions. Qualitative
observations showed that undisturbed specimens contained lamination and segregation of finer and coarser materials, while the reconstituted specimens did not. While the resin-impregnated specimens resulted in some macro-fissuring, the internal core of sample and in fabric appeared to remain preserved. Potential for correlating quantitative XRD data with X-ray micro-CT imaging and particle shape was revealed.
6.0 Summary and Conclusions

Research was conducted with the broad objective of gaining insight into the influence of fabric and microstructure of silts on macroscopic monotonic and cyclic behaviour. The main objective was to develop a technology and methodology for preparing undisturbed and reconstitution specimens for X-ray micro-CT imaging, as well as identify reliable technique(s) for qualitative and quantitative post-processing of those images.

Low-plastic Fraser River Delta silt from one geographic location in the Lower Mainland of British Columbia was used in this research, and its results were compared to other studies on silts from various locations in the same area. The specimens for imaging were prepared from relatively undisturbed thin-walled sharpened-edge tube samples of silt retrieved from a previous geotechnical drilling program. In addition, reconstituted samples of silt were prepared using the method of slurry deposition, and then consolidated to 50, 100, and 200 kPa, were used to observe the effects of various stress states on fabric and microstructure. A calibration sample using glass beads of a known diameter embedded in silt was used to verify the validity of the dataset post-processing analysis.

The following sections summarize the imaging explorations and the findings of this research. Section 6.1 discusses the chosen laboratory methodologies that worked best for specimen sub-sampling and consequently X-ray micro-CT imaging. Section 6.2 summarizes the findings from data acquisition and development of the digital particle segmentation methodology. Section 6.3 presents the quantitative and qualitative results from X-ray micro-CT imaging. A recommended procedure for the sampling, imaging, and data processing of X-ray micro-CT silt specimens is presented in Section 6.4. Finally, Section 6.5 recommends future works for the continuation of this research on the fabric and microstructure of Fraser River Delta silts.

6.1 Laboratory Procedures for X-ray Micro-CT Specimen Preparation

The 7.0-cm diameter stainless-steel tube samples obtained from field drilling program could not be directly used as specimens for imaging of relatively undisturbed silts for a
number of reasons. The higher density of steel tubing compared to the contained material would absorb X-ray energy, resulting in lower quality images. In addition to this, the optimum sample size for imaging was restricted to around 5.0 mm diameter, as larger specimens would require significantly longer scanning times to acquire high resolution images and result in datasets too large for efficient analysis. The specimen size was also restricted as the scanning machine stage can only accommodate small specimens.

With respect to reconstituted specimens, conventional DSS specimens 2.0 cm in height could not be used for this research as the final height of a consolidated sample had to consider disturbance due to the top and base plates during extraction. To ensure that a specimen’s sub-sampled section would be as undisturbed as possible, a taller consolidation cylinder was designed which generated sub-samples a minimum of 4.0 cm in height. This increased height of the specimen ensured that the central core of the sample could be imaged without concern of disturbance from the top and base plates.

Small-diameter (~ 5.0-mm diameter) tubes made of the following three materials were assessed for sub-sampling to obtain specimens from the larger size silt samples: (i) thin-plastic tubes (drinking straws); (ii) sharpened glass tubes; and (iii) 3D-printed tubes. Various inner diameters and wall thicknesses were tested, and the sample tube made of thin-plastic material (drinking straw), which had a B/t ratio of 37.04 was found to be the most successful/effective. The glass tubes had thicker walls and their similar density to the contained material was not ideal for scanning. The walls of the 3D printed tubes were too thick, as their ability to be printed was restricted by the nozzle resolution of the printer. This resulted in little to no recovery during sub-sampling. As such, the thin-plastic drinking straw was the sampling tube of choice for preparation of specimens for X-ray micro-CT.

Once the specimens were extracted from sub-sampling, they had to be sealed prior to shipping for scanning. Paraffin wax and conventional sticky tack were compared for their ability to preserve moisture of the small tube specimens. Sticky tack was shown to be superior in this regard, as the paraffin wax need to be heated prior to sealing and some evaporation of moisture from soil specimens during this process was noted. In
addition to these sealing methods, resin impregnation was attempted for achieving preserved fabric and microstructure of samples. A new-to-practice resin, Henkel Loctite IS 535 RTC, was used due to its room temperature curing qualities and very low viscosity. Various methods of resin impregnation were attempted, and some images showed promising results when comparing non-resin samples to resin samples.

As indicated earlier, the testing program that followed contained samples of various preparation types, including a calibration slurry deposited specimen with glass beads, relatively undisturbed specimens, slurry consolidated specimens to varying consolidation levels, and resin-impregnated samples.

6.2 Scanning and Image Processing Methodology

Once the samples were prepared, they were sent to three different scanning facilities, including: Monash University in Australia, UBC Okanagan in Kelowna, and UBC Pulp and Paper Center (PPC) in Vancouver. ZEISS Xradia 520 Versa scanners were used at Monash and PPC, and an Xradia 400 Versa scanner, an older model, was used at UBC Okanagan. The Xradia 520 scanner at PPC proved to be the most successful scanner at achieving the highest resolution, meaning the smallest voxel size, which was 0.869 µm.

Once the 3D datasets were acquired, their quality was assessed in Avizo 9.7 software. The Median, Symmetric Nearest Neighbour, and Non-Local Means filters were compared for their ability to remove noise from the images while maintaining edges of particles. The Non-Local Means filter proved to be the best choice, confirming recent reviews of the literature.

The appropriate greyscale then had to be chosen for each image to distinguish between solids (more white) and voids (more black). The interactive thresholding tool in Avizo, along with analysis of the valleys in the intensity range histogram of each dataset, allowed for user-chosen greyscale values. The quality of this step depended on whether the images were 8-bit versus 16-bit (16-bit having a wider range of potential grey values).
One of the main requirements for analyzing particle fabric is to digitally segment the particles within the image in order to extract grain characteristics. This required the application of the watershed segmentation algorithm. The validity of this method was shown by glass bead calibration imaging. Glass beads of 1.0 mm diameter were placed in layers in a slurry deposited sample. The material was also sieved between the No. 200 and No. 400 sieves, resulting in known grain diameters between 37 and 75 µm. The image filtering and watershed algorithm as described above were applied to these glass bead specimens. Maximum, intermediate, and minimum diameters ranged from 0.95 to 1.23 mm and 3D calculated volume of the beads was within around 13% of conformance with an idealized volume of a perfect sphere. This confirmed that the methodology was working well, and image processing could proceed for other fabric and microstructure samples.

6.3 Quantitative and Qualitative Results of X-ray Micro-CT Scans

As mentioned previously, a variety of sample types were scanned. Other than the glass bead calibration specimens, the specimen material originated from the same thin-walled, sharped-edge stainless-steel sampling tube, which meant that their grain size distributions would be very similar. To confirm that the post-processing methodology successfully segmented individual grains, a digital grain size distribution, calculated by equivalent volume assuming equal diameter, was compared to laboratory hydrometer grain size curves. Most scanned images conformed to the laboratory-obtained dataset as well as previous datasets on Fraser River Delta silts by other researchers. The highest resolution dataset appeared to over-estimate the fines content of the digital sample, while the lower quality images with higher noise seemed to under-estimate the fines content.

A number of computational biases could have produced the above results. Namely, images that contained the full field of view of the wall showed smearing wall effects along the outer edge of the soil sample closest to the wall (about 1/10th of the diameter into the sample). Soil particles visibility were rearranged parallel to the wall and in a more dense state. This denser state made it more difficult for the lower, 8-bit images to
distinguish multiple greyscales within these regions. In addition to this, the outer edges of the sample are exposed to the highest energy from the X-ray, and since the whole field of view was visible, it was evident that the contrast of the greyscale of the outer edges did not align with the greyscale of the inner core of the samples, making is very difficult to choose the appropriate greyscale. As such, digital aggregations of particles around the edges were created during the segmentation, falsely generating larger particle sizes.

Once the grain size distribution was shown to match well with laboratory data, the void ratio was also compared to laboratory specimens. As the interactive thresholding is highly subjective, accurate void ratios were difficult to obtain. Nonetheless, a few datasets showed promising void ratios around 0.80, which is common for a dense silt.

The development of rose diagrams was also attempted. This was done to note whether the preferential orientation of the longest particle axis would rearrange according to consolidation level. Visually, particle long axes did seem to become more horizontal with increase loading, however computational biases may have resulted in poor data. Some correlations were noted, however cannot be confirmed at this time.

The fabric and microstructure of undisturbed and reconstituted samples was also investigated. Throughout all images, regardless of whether the sample was impregnated with resin, varving or lamination between finer-grained and coarser-grained particles was noted in the undisturbed specimens, but not in the reconstituted specimens. The overall structure and particle arrangement of the reconstituted specimens appears more randomly mixed rather than showing natural lamination. Interestingly in the undisturbed specimens, large void spaces (likely water or decayed organic matter) appeared to occur preferentially within the coarser-grained laminations. Apparent fracturing from resin impregnation also appeared in some coarser-grained areas, however the resin still did appear to preserve the fabric and microstructure of the inner core.

Particle shape characteristics, including roundness, sphericity, and aspect ratio were also calculated in Avizo 9.7. The Fraser River Delta silt particles fell within the spheroid
and roller Zingg shape classification and “very angular” to “angular” as per Power’s grade classes. However, due to difficulty segmenting smaller grains and digital aggregations as described above for wall effects, it is clear that this classification of roundness is not necessarily accurate as the digital aggregations due to similar density of all particles make it difficult to achieve perfectly smooth true shapes. Notes on connections between XRD mineralogical data, particle shape, and density were made to show that X-ray micro-CT, in conjunction with XRD could provide insight on likely particle shapes and overall specimen fabric and microstructure.

Based on the above experimentative findings, the following section provides a recommended protocol to follow for sampling, imaging, and post-processing of X-ray micro-CT datasets gathered for silt-type materials.

6.4 Recommended Methodology for Sampling, Imaging, and Post-Processing

The previous sections have shown that various methods were attempted in each phase of the sampling, imaging, and post-processing procedure of Fraser River Delta silts. The list below provides a general protocol to consider when conducting similar work.

- Prepare undisturbed and reconstituted specimens to a final minimum height of 5.0 cm to reduce possibility for disturbance during top cap removal (for reconstituted specimens) and sub-sampling.
- To sub-sample a specimen, use either a thin-plastic drinking straw or a thin-walled rigid 3D printed tube. Core down vertically, slowly, and consistently with your finger, monitoring if soil is successfully being recovered in the tube.
- Note the moisture content and void ratio of the laboratory specimen.
- Handle specimen with great care, especially those contained within a thin-plastic drinking straw, as the soil can easily be compressed if the tube is pressed with even minimal force.
- Seal specimen at each end with sticky tack, label specimens, wrap with plastic wrap, and place in a moisture-controlled room prior to imaging.
- Perform calibration scans using scanner of choice to ensure proper contrast and resolution can be achieved for the soil under investigation.
• Depending on scanner ability, attempt to achieve a minimum voxel size (resolution) that is at least the size of your minimum grain size of interest. In this case, the ideal minimum resolution was 2.0 µm.

• During imaging setup, treat sample with care, and ensure that it is perfectly vertical in the holding stage.

• For ZEISS Xradia 520 Versa scanners, the 4X objective was sufficient in achieving a high-quality dataset with a resolution less than 1.0 µm.

• For a resolution of around 3.4 µm using a ZEISS Xradia 520 Versa scanner, a suggested starting point for imaging control parameters is as follows:
  o Voltage = 80.2 kV
  o Power = 7.0 W
  o Current = 87.5 µA
  o Optical magnification = 4X
  o Source to object distance = 37.0 mm
  o Detector to object distance = 37.0 mm
  o Exposure time = 2.0 s

• Resolution may be compromised if it is preferred to have the entire specimen (tube wall and contained soil) within the field of view. If this is not an option, then dataset stitching is necessary, which will significantly increase the dataset size.

• A dataset size of 2 gigabytes or greater is common. Ensure that the computer available for post-processing of datasets has a strong 3D graphic card, high computational ability, and sufficient memory.

• Use the Non-Local Means filter for reducing noise while preserving grain edges.

• For interactive thresholding, use the intensity range histogram as a guide to choose the appropriate greyscale cut-off value to distinguish between solid space and pore space.

• 16-bit images (i.e. 65,535 variations of grey tone) are ideal for all aspects of consolidated Fraser River Delta silt specimen imaging, where the specific gravity values of the contained minerals are all very similar.

• Particle segmentation can be successfully performed using the watershed segmentation methodology.
A variety of parameters can be extracted once the analysis is complete, however some essential parameters for quantitative assessment include minimum Feret diameter, intermediate Feret diameter, maximum Feret diameter, 3D volume, 3D surface area, and orientation vector of the longest axis.

In order to consider computational bias, a particle’s calculated diameter should be at least 3 times the image resolution (3 voxels composing the whole volume). A single voxel is not sufficient in representing a true particle shape, size, and volume.

Comparisons of grain size distributions and void ratios are good starting points for comparing digitally acquired data to laboratory data.

This is a preliminary methodology based on the results of this research program to date. As the above investigation has demonstrated, X-ray micro-CT technology has proven to be a suitable approach to study the fabric of soils less than 75 µm in size, namely Fraser River Delta silt. Initial findings show visualizations of individual grains are possible to yield valuable qualitative and quantitative results. Nonetheless, future work is required to further improve and refine the process. Suggestions for these future works are indicated in Section 6.5.

### 6.5 Considerations for Future Works

Below is a list of recommended future works for the application of X-ray micro-CT for visualizing 3D fabric and microstructure of Fraser River Delta silt:

- Thin-plastic drinking straws for sub-sampling proved difficult at times due to the flexibility of the straw. Some specimens were bent and disturbed during the mounting process for scanning preparation. A higher resolution 3D printer showed potential for printing tubes of wall thickness as low as 0.2 mm while still maintaining rigidity and protection for the soil contained within the tube. Sticky tack should be used as the temporary sealing material due to greater moisture preservation however other methods/plugs can be explored.

- Achieving higher resolution (smaller voxel size) scans would allow for more accurate representation of finer-grained particles, namely particles below 10 µm
in size. This allows greater voxels per volume of interest to all particles to achieve more realistic shapes. In addition to this, further investigation on the scanning parameters would benefit the research to ensure that the highest level of density contrast can be achieved between particles, making it easier to distinguish grain boundaries in particle segmentation. 16-bit images should be image type of choice.

- Interactive thresholding based on the user’s choice of greyscale imposes many biases on the subsequent data analysis. Void ratio comparisons should be further explored to ensure digital results match with laboratory data. The analysis algorithm should be further developed to minimize human choice in the greyscale cut-off. The selection of greyscale level can also be calibrated by maintaining the same scanning parameters while scanning the void material and material of known density (such as quartz, $G_s = 2.65$). This would ensure that the same contrast is achieved among all images, resulting in more accurate segmentation.

- Once higher resolutions are achieved and the greyscale thresholding is calibrated, further studies can be performed on specimens of varying stress levels to observe more accurate particle realignment due to consolidation. The rose diagram analysis will likely improve once particles are better delineated from the particle segmentation process.

- Scanning can be done in real-time to observed to effects of compression and extension on the same particle fabric and microstructure. In addition to this, scanning of specimens that have been previously sheared can yield insight into shear-band development, shape, size, and angle.

- Further analysis of particle shape can be done once improved resolution allows for more accurate representation of particle roundness, sphericity, and aspect ratio.

- By conducting the above study on various materials from other depositional environments would allow the researcher to show that the methodologies described throughout this thesis are transferable to other soil types. This would be the beginning of a database of fabric and microstructures and observed macro-behaviours of different risk-adverse soil types.
References


Dassault Systems. (2019). SOLIDWORKS.


MathWorks. (2019). MATLAB.


Appendix – Coding Scripts for Quantitative Data Analysis

A1 – Grain Size Distribution Plot Function

function [Arg1,Arg2,Arg3,Arg4] = GSDPlotFunction(rez,cutoff,M)

%------------------------------------------ GSDPlotFunction ------------------------------------------
% Function for reading in Excel files from Avizo datasets and plotting
% grain size distributions for each dataset using the minimum Feret
% diameter (Width3D) and a cutoff voxel value of 3 times resolution
%------------------------------------------ READ IN EXCEL FILE ------------------------------------------

% 1. Note the last cell in the .csv file obtained from Avizo
% 2. Determine column headings and ensure that they correspond
%    to the assigned matrices below
% 3. Indicate image resolution below in "rez" for removing grain size
%    less than specified cutoff voxel

% Defining columns from Excel
length = M(:,1);
breadth = M(:,2);
thick = M(:,3);
width = M(:,4);
phi = M(:,5);
theta = M(:,6);
area3D = M(:,7);
volume3D = M(:,8);
[M_rows, M_cols] = size(M);

% Determine minimum grain size and remove corresponding data that are
% less than predetermined cutoff range
min_dia1 = min(length, breadth);
min_dia2 = min(thick, width);
min_dia = min(min_dia1, min_dia2);
M = [M(:,1:4) min_dia M(:,5:M_cols)];
remove = M(:,5) < cutoff;
M(remove,:) = [];
[M_rows, M_cols] = size(M);

%---------------- GRAIN SIZE DISTRIBUTION BASED ON VOLUME ----------------

sieve_sizes = [420 354 297 250 210 177 149 125 105 88 74 63 ...
53 44 37 30 20 16 12 8.6 6.1 3.2 1.3 1.0];
[numrows_sievesizes, numcols_sievesizes] = size(sieve_sizes);
M(:,5);
volume = [];
for i = 1:numcols_sievesizes
    k = sum(M(M(:,5)>= sieve_sizes(:,i),9));
    volume = [volume, k];
end
volume.';
total_volume = sum(volume, 'all');

percent_volume = [];
for i = 1
    if volume(i) > 0
        k = 100 - (volume(i)/total_volume*100);
        percent_volume = [percent_volume; k];
    else
        percent_volume = [percent_volume; 100];
    end
end

for i = 2:numcols_sievesizes
    if volume(i-1) > 0
        k = percent_volume(i-1) - (volume(i-1)/total_volume*100);
        percent_volume = [percent_volume; k];
    elseif volume(i-1) == 0
        percent_volume = [percent_volume; percent_volume(i-1)];
    end
end

percent_volume;
sieve_sizes_mm = sieve_sizes./1000;
sieve_sizes_abovecutoff = sieve_sizes(sieve_sizes > cutoff)/1000;
percent_volume_abovecutoff = percent_volume(sieve_sizes > cutoff);

sieve_sizes_belowcutoff1 = sieve_sizes(sieve_sizes < cutoff);
sieve_sizes_belowcutoff = sieve_sizes_belowcutoff1(sieve_sizes_belowcutoff1 >= rez)/1000;
percent_volume_belowcutoff1 = percent_volume(sieve_sizes < cutoff);
percent_volume_belowcutoff = percent_volume_belowcutoff1(sieve_sizes_belowcutoff1 >= rez);

[a,b] = size(percent_volume_abovecutoff);
interpolate_cutoff_percent = mean([percent_volume_abovecutoff(a),...
    percent_volume_belowcutoff(1)]);

percent_volume_abovecutoff = vertcat(percent_volume_abovecutoff,...
    interpolate_cutoff_percent);
sieve_sizes_abovecutoff = horzcat(sieve_sizes_abovecutoff, cutoff/1000);

percent_volume_belowcutoff = vertcat(interpolate_cutoff_percent,...
    percent_volume_belowcutoff);
sieve_sizes_belowcutoff = horzcat(cutoff/1000, sieve_sizes_belowcutoff);

Arg1 = percent_volume_abovecutoff;
Arg2 = sieve_sizes_abovecutoff;
Arg3 = percent_volume_belowcutoff;
Arg4 = sieve_sizes_belowcutoff;
end
A2 – Plotting of Grain Size Distribution Data

clc; clear all; close all; format short;

%----------------- GRAIN SIZE DISTRIBUTION PLOTTING CODE -----------------

%----------------- REFER TO GSDPlotFunction FOR ALL DATASETS ----------------

% GSD analysis for UD2 Resin UBCO Full
rez = 3.2;
cutoff = rez*3;
M = readmatrix('UD2-in-resin-UBCO FULL Label-Analysis', 'Range', 'A3:L531236');

[percent_volume_abovecutoff, sieve_sizes_abovecutoff, ...  
    percent_volume_belowcutoff, sieve_sizes_belowcutoff] ...  
    = GSDPlotFunction(rez,cutoff,M);

a1 = percent_volume_abovecutoff;
b1 = sieve_sizes_abovecutoff;
c1 = percent_volume_belowcutoff;
d1 = sieve_sizes_belowcutoff;

% GSD analysis for UD2 Monash
rez = 3.38;
cutoff = rez*3;
M = readmatrix('UD2 Monash Full Label Analysis v2 11oct2019', 'Range', 'A3:L605590');

[percent_volume_abovecutoff, sieve_sizes_abovecutoff, ...  
    percent_volume_belowcutoff, sieve_sizes_belowcutoff] ...  
    = GSDPlotFunction(rez,cutoff,M);

a2 = percent_volume_abovecutoff;
b2 = sieve_sizes_abovecutoff;
c2 = percent_volume_belowcutoff;
d2 = sieve_sizes_belowcutoff;

% GSD analysis for FR200 PPC
rez = 0.869;
cutoff = rez*3;
M = readmatrix('FR200Martinez.Label-Analysis.csv', 'Range', 'A3:L81434');

[percent_volume_abovecutoff, sieve_sizes_abovecutoff, ...  
    percent_volume_belowcutoff, sieve_sizes_belowcutoff] ...  
    = GSDPlotFunction(rez,cutoff,M);

a3 = percent_volume_abovecutoff;
b3 = sieve_sizes_abovecutoff;
c3 = percent_volume_belowcutoff;
d3 = sieve_sizes_belowcutoff;

% GSD analysis for FR50 UBCO
rez = 3.2;
cutoff = rez*3;
M = readmatrix('FR50-UBCO.Label-Analysis', 'Range', 'A3:L618993');
a4 = percent_volume_abovecutoff;
b4 = sieve_sizes_abovecutoff;
c4 = percent_volume_belowcutoff;
d4 = sieve_sizes_belowcutoff;

% GSD analysis for UBCO 1B-2
rez = 3.2;
cutoff = rez*3;
M = readmatrix('UBCO-1B-2.Label-Analysis','Range', 'A3:L761701');

a5 = percent_volume_abovecutoff;
b5 = sieve_sizes_abovecutoff;
c5 = percent_volume_belowcutoff;
d5 = sieve_sizes_belowcutoff;

% Historical UBC research lab data
lab_x = [0.42, 0.297, 0.149, 0.105, 0.075, 0.04101, 0.0195, ...
0.01602, 0.01203, 0.00857, 0.00616, 0.00315, 0.00130];
lab_y = [100.00, 100, 100, 94, 80.2, 74.26, 67.19, 56.58, 44.20, 35.36, ...
31.83, 26.52, 17.68, 14.15];

sanin_x = [0.425, 0.3, 0.15, 0.106, 0.075, 0.05, 0.035, 0.025, 0.017, ...
0.0105, 0.0085, 0.006, 0.0045, 0.0028, 0.0011];
sanin_y = [100 100 99 94 90 82, 72, 61, 46, 33, 25, 20, 16, 12.5, 8];

soysa_x = [0.42 0.25 0.21 0.177 0.149 0.105 0.074 0.063...
0.044 0.01977 0.01551 0.01318 0.01200 0.01071 0.00878 0.00736...
0.00544 0.00395 0.00288 0.00222 0.00130 0.00090 0.00082];
soysa_y_lower = [98.97 96.57 95.23 93.72 91.43 83.46 76.23...
70.32 63.91 54.03 48.29 46.66 41.84 39.96 37.13...
34.31 27.72 25.84 23.01 20.19 16.42 14.54 13.60];
soysa_y_upper = [99.24 97.05 96.42 95.95 95.46 94.09 86.11...
80.34 65.46 54.66 49.98 47.18 45.30 42.54 40.63...
37.76 32.97 29.14 25.31 22.44 19.12 16.31 15.38];

verma_x_siteA = [0.15 0.075 0.021 0.0075 0.0055 0.003 0.0011];
verma_y_siteA = [100 98 60 38 32 20 12];

verma_x_siteE = [0.25 0.15 0.106 0.075 0.05 0.035 0.029 0.025 0.0205...
0.019 0.011 0.0105 0.0085 0.007 0.0061 0.00305 0.0011];
verma_y_siteE = [100 100 98 95 86 76 70 64 60 58 46 40 32 30 28 19 10];

%----------------- PLOT ALL GRAIN SIZE DISTRIBUTION -----------------

figure(1)
hold on
silt = [0.002,0.002];
sand = [0.075,0.075];
gravel = [4.75, 4.75];
range = [0, 100];
plot(silt, range, 'Color', 'k', 'LineWidth', 0.05)
plot(sand, range, 'Color', 'k', 'LineWidth', 0.05)
plot(gravel, range, 'Color', 'k', 'LineWidth', 0.05)
box on

curve1 = plot(sanin_x, sanin_y, ...
    'Color', 'k', ...
    'LineWidth', 0.5, ...
    'LineStyle', ':', ...
    'Marker', 'x', ...
    'MarkerSize', 4);
curve2 = plot(soysa_x, soysa_y_lower, ...
    'Color', 'k', ...
    'LineWidth', 0.5, ...
    'LineStyle', ':', ...
    'Marker', 'v', ...
    'MarkerSize', 4);
curve3 = plot(verma_x_siteA, verma_y_siteA, ...
    'Color', 'k', ...
    'LineWidth', 0.5, ...
    'LineStyle', ':', ...
    'Marker', '^', ...
    'MarkerSize', 4);
curve4 = plot(verma_x_siteE, verma_y_siteE, ...
    'Color', 'k', ...
    'LineWidth', 0.5, ...
    'LineStyle', ':', ...
    'Marker', '(', ...
    'MarkerSize', 4);
curve5 = plot(soysa_x, soysa_y_upper, ...
    'Color', 'k', ...
    'LineWidth', 0.5, ...
    'LineStyle', ':', ...
    'Marker', 'd', ...
    'MarkerSize', 4);

% Curve 6: Laboratory
curve6 = plot(lab_x, lab_y, ...
    'Color', 'r', ...
    'LineWidth', 1.0, ...
    'Marker', 's', ...
    'MarkerEdgeColor', 'k', ...
    'MarkerFaceColor', 'r', ...
    'MarkerSize', 8);

% Curve 7/8: UD2 in resin UBCO
curve7 = plot(b1, a1, ...
    'Color', 'b', ...
    'LineWidth', 1.0, ...
    'Marker', '^', ...
    'MarkerEdgeColor', 'k', ...
    'MarkerFaceColor', 'b', ...
    'MarkerSize', 7);
curve8 = plot(d1, c1, ...
    'Color', 'b', ...
'LineWidth', 1.0, ...
'LineStyle', '--', ...
'Marker', '^', ...
'MarkerEdgeColor', 'b', ...
'MarkerSize', 7);

% Curve 9/10: UD2 Monash
curve9 = plot(b2, a2, ...
'Color', 'g', ...
'LineWidth', 1.0, ...
'Marker', 'v', ...
'MarkerEdgeColor', 'k', ...
'MarkerFaceColor', 'g', ...
'MarkerSize', 7);

curve10 = plot(d2, c2, ...
'Color', 'g', ...
'LineWidth', 1.0, ...
'LineStyle', '--', ...
'Marker', 'v', ...
'MarkerEdgeColor', 'g', ...
'MarkerSize', 7);

% Curve 11/12: FR200 PPC
curve11 = plot(b3, a3, ...
'Color', 'm', ...
'LineWidth', 1.0, ...
'Marker', 'd', ...
'MarkerEdgeColor', 'k', ...
'MarkerFaceColor', 'm', ...
'MarkerSize', 7);

curve12 = plot(d3, c3, ...
'Color', 'm', ...
'LineWidth', 1.0, ...
'LineStyle', '--', ...
'Marker', 'd', ...
'MarkerEdgeColor', 'm', ...
'MarkerSize', 7);

% Curve 13/14: FR50 UBCO
curve13 = plot(b4, a4, ...
'Color', 'y', ...
'LineWidth', 1.0, ...
'Marker', 'o', ...
'MarkerEdgeColor', 'k', ...
'MarkerFaceColor', 'y', ...
'MarkerSize', 7);

curve14 = plot(d4, c4, ...
'Color', 'y', ...
'LineWidth', 1.0, ...
'LineStyle', '--', ...
'Marker', 'o', ...
'MarkerEdgeColor', 'y', ...
'MarkerSize', 7);

% Curve 15/16: UBCO 1B-2
curve15 = plot(b5, a5, ...
'Color', 'c', ...

curve16 = plot(d5, c5, ...
'Color', 'c', ...
'LineWidth', 1.0, ...
'LineStyle', '--', ...
'Marker', 'o', ...
'MarkerEdgeColor', 'c', ...
'MarkerSize', 7);
'LineWidth', 1.0, ...
'Marker', 's', ...
'MarkerEdgeColor', 'k', ...
'MarkerFaceColor', 'c', ...
'MarkerSize', 7);
curve16 = plot(d5, c5, ...
'Color', 'c', ...
'LineWidth', 1.0, ...
'LineStyle', '-', ...
'Marker', 's', ...
'MarkerEdgeColor', 'c', ...
'MarkerSize', 7);

%------------------- LABEL GRAIN SIZE DISTRIBUTIONS -----------------------
set(gca, 'FontSize', 14)
xlim([0.001, 100])
ylim([0, 100])
xlabel('Diameter (mm)')
ylabel('Percent Passing by Mass Equivalent Volume (%)')
set(gca, 'XScale', 'log', 'xticklabels', xaxis);
the_legend = legend([curve6, curve7, curve9, curve11, curve13, curve15, ...
                      curve1, curve2, curve3, curve4, curve5], ...
                      'Laboratory (present study)', 'UBCO-FR-UD-StrRes', 'MON-UD-FR-Str', ...
                      'PPC-FR-200', 'UBCO-FR-50', 'UBCO-FR-SD-100-Str', '...
                      'Sanin (2010) - Average', '...
                      'Position', [0.75 0.5, 0.1 0.1]);
title(the_legend, 'LEGEND');

%---------------------- PLOT SOIL TYPE DISTINCTION ------------------------
dim = [0.104 0.81 0.1 0.1];
str = 'CLAY';
annotation('textbox', dim, 'String', str, 'FitBoxToText', 'on', 'FontWeight', '...
          'bold', 'LineWidth', 1.0, 'BackgroundColor', 'w', 'HorizontalAlignment', 'center', 'FontSize', 10);
dim = [0.242 0.81 0.1 0.1];
str = 'SILT';
annotation('textbox', dim, 'String', str, 'FitBoxToText', 'on', 'FontWeight', '...
          'bold', 'LineWidth', 1.0, 'BackgroundColor', 'w', 'HorizontalAlignment', 'center', 'FontSize', 10);
dim = [0.517 0.81 0.1 0.1];
str = 'SAND';
annotation('textbox', dim, 'String', str, 'FitBoxToText', 'on', 'FontWeight', '...
          'bold', 'LineWidth', 1.0, 'BackgroundColor', 'w', 'HorizontalAlignment', 'center', 'FontSize', 10);
dim = [0.758 0.81 0.1 0.1];
str = 'GRAVEL';
annotation('textbox', dim, 'String', str, 'FitBoxToText', 'on', 'FontWeight', '...
          'bold', 'LineWidth', 1.0, 'BackgroundColor', 'w', 'HorizontalAlignment', 'center', 'FontSize', 10);
A3 – Rose Diagram Plotting

clc; clear all; close all; format short;

%---------------------------- CREATION OF ROSE DIAGRAM PLOTS -----------------------------------

% This script is for the creation of rose diagrams (cumulative angle alignment polar plots) from Avizo data on the deviation angles of individual particles. Their axis of reference is adapted to indicate zero degrees as horizontal. The Excel reading-in process is the same as the GSDPlotFunction process.

% Read-in Excel file and assign columns as variables
rez = 3.2;
cutoff = rez*3;
M = readmatrix('FR50-UBCO.Label-Analysis','Range', 'A3:L618993');
length = M(:,1);
breadth = M(:,2);
thick = M(:,3);
width = M(:,4);
phi = M(:,5);
theta = M(:,6);
area3D = M(:,7);
volume3D = M(:,8);
[M_rows, M_cols] = size(M);

% Determine minimum grain size and remove corresponding data that are less than predetermined cutoff range
min_dia1 = min(length, breadth);
min_dia2 = min(thick, width);
min_dia = min(min_dia1, min_dia2);
M = [M(:,1:4) min_dia M(:,5:M_cols)];
remove = M(:,5) < cutoff;
M(remove,:) = [];
[M_rows, M_cols] = size(M);

% Redefine columns, excluding cutoff values
length = M(:,1);
breadth = M(:,2);
thick = M(:,3);
width = M(:,4);
phi = M(:,5);
theta = M(:,6);
area3D = M(:,7);
volume3D = M(:,8);

%-------------------------------- ROSE DIAGRAM -----------------------------------

[numrowsM, numcolsM] = size(M);
rose = [];
for i = 1:numrowsM
    if theta(i) > 0 & theta(i) < 90;
        newphi = 90-phi(i);
        rose = [rose, newphi];
    elseif theta(i) >= 90;

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newphi = 90+phi(i);
rose = [rose, newphi];
elseif theta(i) < 0 & theta(i) >= -90;
newphi = 90+phi(i);
rose = [rose, newphi];
elseif theta(i) < -90;
newphi = 90-phi(i);
rose = [rose, newphi];
end

rose.

angles = [0 15 30 45 60 75 90 105 120 135 150 165 180];
angles_count = histcounts(rose, angles)
edges = [0 pi/12 pi/6 pi/4 pi/3 5*pi/12 pi/2 7*pi/12 2*pi/3 3*pi/4 ...
5*pi/6 11*pi/12 pi];

figure(1)
polarhistogram('BinEdges', edges, 'BinCounts', angles_count, ...
'FaceAlpha', 0.4)
thetatickformat('degrees')
thetalim([0 180])
title('Principal Axis Orientation Diagram (\phi) for UBCO-FR-50')