

**THREE-DIMENSIONAL PRINTING OF LEAN
CLAY CERAMICS**

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THREE-DIMENSIONAL PRINTING OF LEAN CLAY CERAMICS

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**A project report submitted in partial fulfilment of the
requirements for the award of Bachelor of Engineering
(Honours) Mechanical Engineering**

**Lee Kong Chian Faculty of Engineering and Science
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May 2022

DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

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ABSTRACT

3D printing is an additive manufacturing technology in which the material is laid down layer by layer to form a three-dimensional object. There are various materials which can be employed in this technology such as thermoplastic, metal, and clay. In the past decades, various research has been carried out on the 3D printing of polymers and metallic materials; however, the research on clay ceramic is still limited. In this research, lean clay is used as the raw material for the 3D printing using Direct Ink Writing (DIW) technique. The main aim of this project is to investigate the relationship between the clay type with different moisture content, the rheological properties, and the optimal printing criterion. Herein, the lean clays were prepared with moisture content ranging from 30% to 47%. The rheological properties of the clays were studied using a rheometer. The relationships between the moisture content, rheological behaviour, and print parameters were evaluated. The effect of different water content of the lean clay on its printability through different nozzle sizes was also investigated. Various printing tests have been carried out to access the optimal printing criterion. From the results of the rheology test, it can be deduced that the viscosity of the clay decreases as the shear rate increases regardless of the moisture content. Besides, the storage modulus and yield stress of the clay samples increase as the water content decreases. Moreover, it was discovered that frequency possesses minimal effect on the storage modulus of the clay samples. On the other hand, the results of printing tests denote that moisture content ranging from 40% to 47% produces the clay suspensions that are printable. Moreover, it was also discovered that clogging is deemed to have occurred for the nozzle diameter below 1.20 mm (16G). Ultimately, this project has demonstrated the feasibility of lean clay as the raw material of ceramic printing.

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LIST OF SYMBOLS / ABBREVIATIONS

3D	Three dimensional
FDM	Fused Deposition Modelling
SLA	Stereolithography
SLS	Selective Laser Sintering
LDM	Liquid Deposition Modelling
AM	Additive Manufacturing
DIW	Direct Ink Writing
L/D	Height to base diameter
D	Diameter
w	water content, %
M_{ems}	mass of container and moist specimen, g
$M_{c ds}$	mass of container and oven dry specimen, g
M_c	mass of container, g
M_w	mass of water, g
M_s	mass of oven dry specimen, g
τ	shear stress, Pa
τ_0	yield stress, Pa
k	consistency index, Pa·s ⁿ
$\dot{\gamma}$	shear rate, s ⁻¹
n	flow index

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CHAPTER 1

INTRODUCTION

1.1 General Introduction

3D printing is an additive manufacturing technology in which the material is laid down layer by layer to form a three-dimensional object. Generally, 3D printing is used to create three-dimensional solid objects through the integration of processes and technologies such as additive manufacturing, thin horizontal cross-sections, material selection, and computer programming. With the advancement of 3D printing, mankind can eventually create almost anything such as utensils, tools, machining parts, etc. Herein, the role of 3D printing has induced vast changes in the entire industry, which is helpful for any organisation, individual, etc., while becoming more and more available to the public.

In the past decades, there has been various research on the 3D printing of polymers and metallic materials; however, the research on clay ceramics is still limited. In general, polymers such as thermoplastics are simpler to print because they solidify at room temperature after heating, and they offer no collapse problem. On the other hand, when dealing with fluid-dense ceramics material such as clay through the printing process, there are few limitations due to geometry, collapse, drying, and retirement. Due to such limitations, 3D printing of clay ceramics remains an open challenge. In this context, this project intends to explore the printability of lean clay through Direct Ink Writing (DIW) technique. In DIW, the material properties of the extruded material play a fundamental role in determining its printability. Herein, a typical 3D printing method divides an object into thousands of small slices and then builds it up from the bottom up, slice by slice (Shahrubudin et al.2019). The microscopic layers adhere to one another to produce a solid entity. Hence, the material properties such as rheological behaviour of the lean clay should be considered before printing.

1.2 Importance of the Study

In today's world, the application of 3D printing technology has seen a noticeable rise in various industries in conjunction with Industry Revolution 4.0. The reason for the overnight rise in the popularity of 3D printing is the increasing accessibility which is made possible by a variety of factors. Herein, the cost of 3D printers has come down significantly over the years which seems to be very helpful to its market growth. Besides, 3D printing technology has been widely adopted due to its potential in realising the creative idea of the consumers. In this context, 3D printing technology plays an important role in this era of modernization as it possesses the potential to replace consumerism, which can eventually empower the people to make the unprecedented customization of their own goods. As many people believe, any technology in the world is here to stay, and with time it continues and becomes more common which is further to be refined and evolved.

The 3D printing technique used in this project involved turning aqueous clay paste into solid structure via Direct Ink Writing (DIW) technique. DIW is a cost-effective technology that is adopted by the ceramic industry as well as the creative community. The material used in this project is lean clay which is rarely explored by other researchers. Hence, the importance of this project is to explore a new material in the 3D printing of clay ceramics. Herein, the clay can be made with various moisture content and its printability will be accessed. This project has introduced a new raw material to be used in 3D printing. In short, the importance of the study is to increase the future progress of ceramics printing with lean clay as raw material.

1.3 Problem Statement

Nowadays, the 3D printing of lean clay ceramics is rarely explored by other researchers or companies. Various research has been carried out on the commercial clay which are commonly used in the ceramic industry. In this context, more materials should be explored to improve the versatility of the raw material in ceramic printing. With this, a wider range of material can be adopted in the 3D printing of clay ceramics.

In addition, there will always be an important question whether the 3D printing technology is able to help the researchers or manufacturers to create

their desired prints on the products with a variety of materials. Hence, this project explores the feasibility of the lean clay to be used as the raw material of 3D printing of ceramics. Herein, clay samples are prepared with different water formulations and their rheological properties of lean clay are studied. Besides, the effect of rheology on the printability of lean clay has been investigated. Various printing has been developed to access the printing criterion of lean clay.

1.4 Aim and Objectives

In relation to the problem statement, the main aim of this project is to investigate the relationship between the clay type with different moisture content, the rheological properties, and the suitable printing criterion. In order to achieve the main aim of this project, several objectives have been defined:

- (i) To study the rheological behaviour of different water content in lean clay and correlate the rheology behaviour with the printability of the clay.
- (ii) To evaluate the print results from different nozzle sizes.
- (iii) To optimise the printing parameter for lean clay.

1.5 Scope and Limitation of the Study

The scope of this study will be focusing on the 3D printing of the lean clay ceramics. Lean clay is selected as the only raw material of this project. Besides, the moisture content of the clay samples tested in this project are ranging from 30% to 47%. Moreover, four nozzle diameters are used for the printing which include 0.60 mm (20G), 0.84 mm (18G), 1.20 mm (16G), and 1.55 mm (14G).

In addition, the physical behaviour and the chemical behaviour of material are the characteristics that help people to create 3D printing products. With a better understanding of the material characteristics of the raw material, it would be beneficial in building or creating high-quality products. Hence, it is vital to understand the chemical composition of lean clay. However, it requires a high level of chemical knowledge to be able to fully comprehend the chemical behaviour of the clay. Therefore, the insufficient knowledge in the chemical composition of the clay is a limitation of the study.

1.6 Contribution of the Study

The main contribution of the study is that it will increase the versatility of raw material that can be used in the 3D printing of clay ceramics. Thus, it reduces the limitation of the materials for producing different types of products with 3D printers. From the viewpoint of engineers, it helps to open more possibilities for future research to provide information about the relationships between the material properties, process and parameters of printing which require close consideration. From the viewpoint of the consumers, this project enhances the potential to create new printing products. Herein, the raw material used for 3D printing in the ceramics industry can also be expanded.

1.7 Outline of the Report

This report covers the study on the 3D printing of lean clay ceramics. A brief introduction about the topic has been covered in Chapter 1. Next, Chapter 2 summarises the literature review which is related to this project. Detailed planning of the methodology and work plan is being discussed in Chapter 3. Then, the results and findings of this project are being presented and discussed in Chapter 4. Finally, Chapter 5 covers the conclusions and recommendations in this project.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In general, 3D printing is a type of additive manufacturing, which involves mixing layers of material from a CAD file to create a three-dimensional object. As new technologies and materials to construct with are developed, manufacturing and engineering are resetting the boundaries of what is possible. So, now that the industries are entering the fourth Industrial Revolution, 3D printing is becoming increasingly popular. However, one question constantly arises such that whether the technology is referred to as 3D printing or additive manufacturing. In this context, there is no correct or incorrect response to this question. In theory, they are both describing the same process. However, there are numerous situations and settings in which one word is preferred over another, and it all comes down to scale and accuracy. Typically, additive manufacturing is used to describe complex and more precise technologies such as SLS, whereas 3D printing is more commonly used to represent 'entry level' processes such as FDM. It makes no difference since 3D printing is a type of additive manufacturing, and everything manufactured using additive manufacturing is 3D printed. Herein, the 3D printing technology involves using software to create the design of 3D objects that will be produced by adding layer by layer of the raw material. Several materials such as clay, plastic, and metal can serve as the raw material of 3D printing.

2.2 Additive Manufacturing

Additive manufacturing, in its simplest form, is an industrial-scale 3D printing that encompasses a wide range of sophisticated processes such as selective laser sintering (SLS). Sintering is the method which involves laser sintering of powder material at a computer-defined location utilising 3D design to build a hard mass of materials by warming way beyond the point of decomposition. This kind of process will provide a more accurate and professional finished product such that it is much harder to see the layers of the building. In general,

AM provides a vast range of results with great accuracy and a wide range of varieties.

2.2.1 Application of Additive Manufacturing

3D printing's earliest applications were for rapid prototyping. Stereolithography offered designers and engineers a faster, more digital route to design a product based on easy iteration. While the technology still has a stronghold in prototyping, additive manufacturing (AM) has advanced far beyond this with the maturation of various technologies and expansion of its materials envelope. Besides, the ability of AM to quickly build complex geometries has made it popular for tooling applications.

3D printing can also be used to build tools that directly or indirectly form the geometry of a final production piece, for instance the tooling for composites layup, thermoforming, vacuum forming, casting (both sacrificial patterns and sand tools themselves), sheet metal bending and injection moulding. In some cases, 3D printing brings additional benefits to the production process, as in the case with conformal cooling channels in injection moulds which enables more efficient cooling, leading to faster cycle times and improved part quality.

2.3 Classification of Clay: Lean Clay

Classification is the arrangement of clay/soil into different groups based on its specific characteristics, properties, and limitations. This classification has a variety of advantages such as selecting a suitable foundation for a specific structure or increasing crop production (ASTM D2487, 2017).

In general, the most troublesome soil in engineering is the one that contains a large proportion of silts and clays because these soils exhibit large variation in their physical properties upon a change in their moisture content (Srivastava *et al*, 2021). The clay soil may resist heavy loads when dry but when become it becomes wet, it will turn into a swamp (quagmire). Thus, these soil shrinks and expands upon a decrease or increase in its moisture content respectively (UKEssays, 2018). One such type of clay soil which contains a large content of silt or sand is called lean clay (ASTM Standard D2487, 2017). These clays contain minerals that absorb a small quantity of water and undergo

very little volumetric change, when wet. Due to the high percentage of silt or sand, this type of soil has very poor compressibility and water stability (Casagrande, A. and M. ASCE., 1948).

The clay/soil classification was formally introduced in 1952 when Prof. Arthur Casagrande of Harvard university agreed with U.S corps of Engineers and Reclamation Bureau and modified his “Airfield Classification System” into the “Unified Soil Classification System” (Srivastava *et al*, 2021). In 1984 the American Society for Testing and Material (ASTM) used the USCS as the base and standardised the soil classification system. Since the various systems such as USDA textural classification System, AASHTO classification system, and Unified soil classification system, are used commonly throughout the world, to classify clay/soil into different groups (ASTM D2487, 2017). Based upon this classification, the clays are then classified into fat clays and lean clays (Srivastava *et al*, 2021).

According to ASTM D2487 (2017), the fine-grained soils whose percent passing through sieve # 200 is 50% or more and the liquid limit of which is less than 50, containing inorganic content, is classified as lean clay. These clays are denoted by CL in which C represents “clay” and L stands for “Low plasticity” (ASTM D2487, 2017).

The liquid limit of the clay soil plays a direct role in its compressibility (more the liquid limit more will be the compressibility). Therefore, the USCS classification system is used for clays which consider the liquid limit as a characteristic parameter between low compressible (lean clays) and high compressibility clays.

The lean clays have a very smooth texture because of their smaller particle size as compared to granular soils, which results in high density and bonds its particles together. Depending upon the content of silt or sand, the clay can further be subdivided into classes such as granular clay, sandy clay, silty clay (lean clays), and heavy clay (Casagrande, A. and M. ASCE., 1948). The clay which has a high content of sand or silt ranging from low to medium plasticity are lean clays.

The consistency of lean clays may be very soft (viscous liquid) or hard (as solid) depending on its moisture content. The clay soil holds more water than most of the other soil types and therefore is used for cultivation and crop

production. Furthermore, the lean clays can be stabilised with mechanical or chemical stabilisation techniques, to improve their strength for constructing highways or building infrastructures.

Generally, the clay soil has very little resistance to deformation when in wet conditions but upon drying becomes a hard mass. That is why clay soil is very difficult to compact in wet conditions and the drainage of water by ordinary means is almost impossible. The classification system developed by AASHTO and ASTM (which is mainly USCS classification) is contemporarily used throughout the world. However, for classifying the nature of the soil, corresponding laboratory tests such as gradation test (particle size distribution), plastic limit, and liquid limit should be performed according to relevant standards (Casagrande, A. and M. ASCE., 1948).

2.4 3D Printing of Clay Ceramics

Historically, when we talk about the manufacturing of ceramic materials, the most commonly used materials are clays. This is because they are relatively easy to find in any type of environmental condition due to the fact that they are natural material. Herein, with the evolution of materials, more possibilities of manufacturing have arrived. There are various types of ceramics materials which have seen their usage in domestic purposes like crockery, and in the construction field like bricks.

3D printing of clay ceramics is a fairly broad term which includes every possible thing between ceramics and alumina. Ceramics may be traced back to Greece, where the clay was fired at high temperatures to solidify it. Ceramics are solid materials made up of metals, non-metals, or an inorganic compound with ionic and covalent bonding, according to technology. From that perspective, carbon and silicon will be considered ceramics, which is crucial to highlight because many 3D printable ceramics have names that seem like metals because they are not made of clay. Ceramics are currently divided into two categories: classical ceramics, which are created entirely of natural raw materials (clay), and technological ceramics, which incorporate silicon, carbon, and nitrogen.

Classical ceramics include stoneware, pottery, and dinnerware, whereas technical ceramics are referred to as engineering ceramics and commercial ceramics, and the list could go on and on since they are frequently

created as tailored issue solutions for specific uses. Aluminium nitride, zirconium, silicon nitride, silicon carbide, and alumina are a few examples of popular technical ceramics. When compared to traditional ceramics, technical ceramics will significantly improve structural, thermal, chemical, and electrical qualities. The majority of 3D printed ceramics are classified as technical, although extrusion, which is based on the screen printing, is mostly used with traditional ceramics.

In this part, an overview of various 3D printing technologies such as SLA, LDM, and DIW will be discussed. Herein, the most used technique in the 3D printing of clay ceramics is DIW, which is a form of additive manufacturing technique that involves building up material layer by layer. Figure 2.1 shows the printing process of clay ceramic using DIW technique.

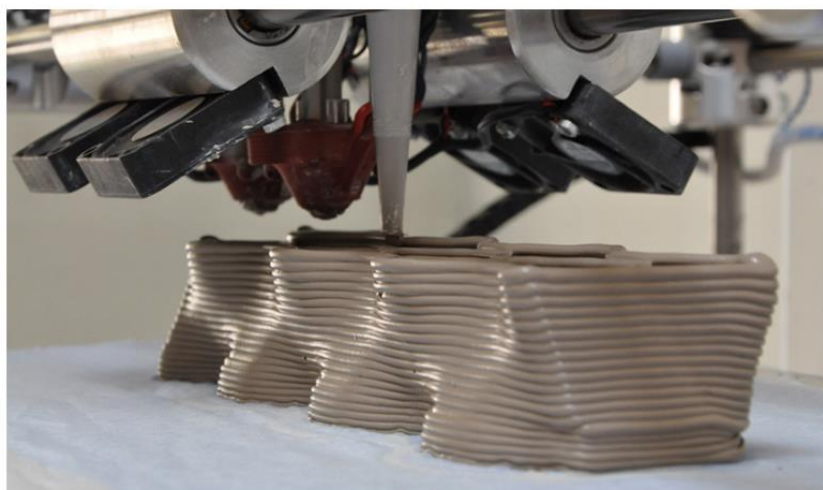


Figure 2.1: Printing of Clay Ceramics Using DIW Technique (Carlota, 2019).

2.4.1 SLA (Stereolithography)

In SLA process, a liquid suspension of fine grain ceramics powder and a UV-sensitive monomer creates layers by layers to form green bodies which are then cured by irradiation with a DLP-projector. Then, the green bodies will undergo multi-stage thermal treatments at firing temperatures up to 1,600 °C. Polymer will be used as binder which will be removed during the removal phases. A final sintering process will give the printed component the distinctive characteristics of ceramics. Figure 2.2 displays the setup of SLA process.

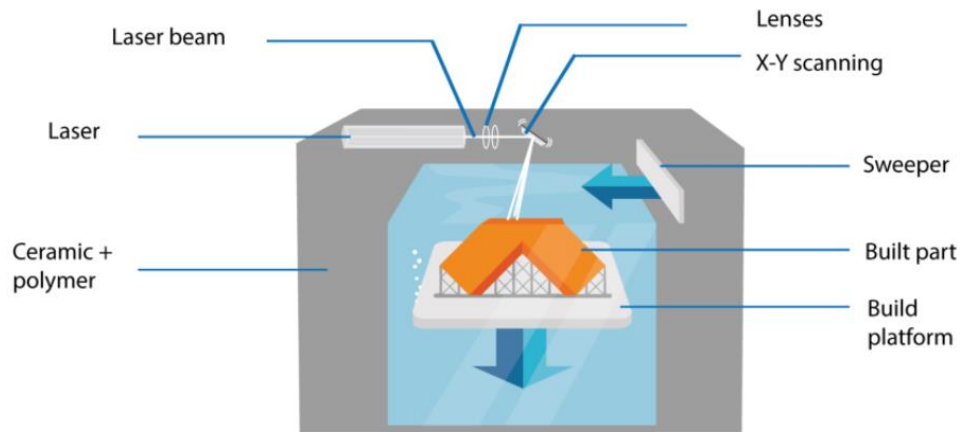


Figure 2.2: Setup of SLA Process (Hernandez, 2020).

2.4.2 LDM (Liquid Deposition Modelling)

The wasp extruders for liquid deposition modelling are pneumatic devices in which a pump delivers the paste pottery material to the installation arm. Perfect combination of a screw extruder and a pressure extruder, it may achieve accuracy that is extremely near to that of a plastic polymer extrusion process. It is feasible to carefully regulate the flow of material using this technology, as well as to employ retreats to break the deposition. A technology that removes air bubbles in the mixture, as well as screw extruders with an external air amplifier of up to 40 bars, are among the innovations. Figure 2.3 shows the setup of LDM process.

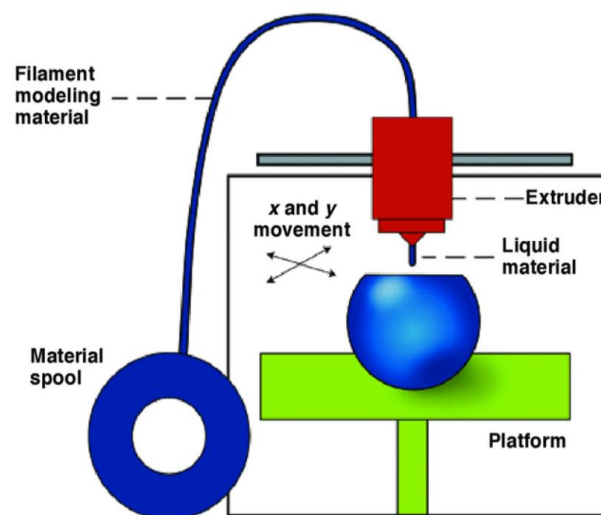


Figure 2.3: Setup of LDM Process (Growth *et al*, 2014)

2.4.3 DIW (Direct Ink Writing)

The 3D printing technique used to create 3D structures, for which the materials (processed as paste or pellets) are deposited in a layer-wise method and by the continuous rising of the print head, is known as direct ink writing technique (DIW).

Over the past 30 years, new technologies are developed for the rapid processing of materials in an economical way and by joining complex 3D parts, commonly termed as 3-dimensional technologies. In 2015 ASTM along with the international organisation for standardisation technical committee, together released a new standard ISO/ASTM 52900:2015 in which the terms, standards, and definitions regarding 3D printing technology were established (Solís Pinargote *et al*, 2020).

The major types of 3D printing that are commonly used are vat photopolymerization, material jetting, and material extrusion. Among them the most common is vat photopolymerization. All these techniques work on the basic layer by layer 3D printing technique but the material processing for each process is different respectively (Solís Pinargote *et al*, 2020).

The introduction of the direct ink writing technique proposes a powerful method for constructing complex 3D parts. It also offers great versatility and effective suitability for the fabrication of ceramic parts. This method is also referred to as robocasting (An extrusion-based technique used to implement new materials most economically and flexibly) by some researchers (Solís Pinargote *et al*, 2020).

The main requirements of this technology include the usage of pastes, capable of maintaining their position and shape and does not allow any collapse during the 3D printing process. The other requirements include correct selection of number of components, additives, and solid-state parameters.

The additives used in the material processing are continuously developing and aimed to produce paste with any aqueous solution. The DIW technology has been on an uprise since its introduction in the 1970. Since then, many powder based, slurry based, and bulk solid based techniques have been developed. In short, direct ink writing is not yet fully developed and has a large room for extensive research and critical solutions.

2.5 CAD for 3D Printing of Clay Ceramics

2.5.1 Layer Height

Layer height is an important parameter to be considered in printing. Herein, before considering the design of the printed model, it is vital to take into account the size of the nozzle and the height of the respective layer. If the layer by layer is too high in relation to the nozzle, then the clay will not be able to stick well together. Meanwhile, if the layer height is too small, it may encounter the risk of over-extruding due to pushing too much clay on top of each other, which may affect prints to pop off or wear out. Therefore, it is important to understand the relationship between layer height and nozzle diameter so that an appropriate printing can be achieved. Figure 2.4 shows a guide on selecting the layer heights based on the nozzle sizes.

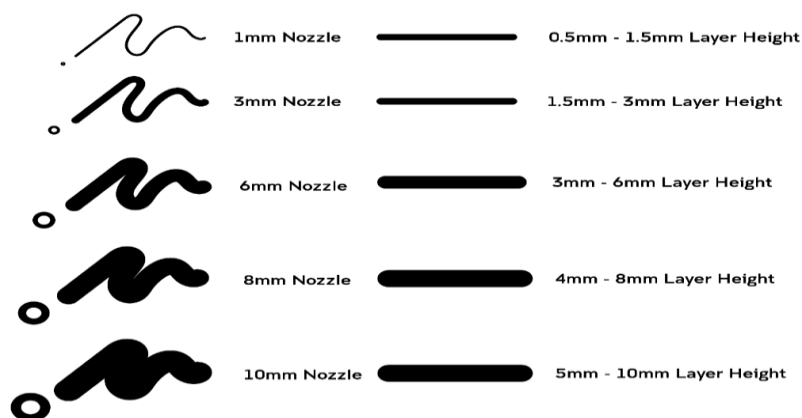


Figure 2.4: Guideline on Selecting the Layer Heights Based on the Nozzle Sizes (UNSW Making, n.d.).

2.5.2 File Preparation

It is a typical process for a 3D printer where a STL file is generated to enable the communication between the CAM software with the 3D printer. This type of file would commonly be generated in 3D modelling programs such as Solidworks.

2.5.3 Slicing

When the STL file is ready, then it will be cut into layers or slices as a printing plan for the 3D printer. The slicing program will generate a new file type known

as G-code. G-codes are generally sets of instructions that tell the printer where to move and how many particles of clay to remove or extrusion. Eventually, the file will be loaded out into the clay printer and used to create the 3D models.

2.6 Design Considerations in Ceramic Printing

2.6.1 Wall Thickness

Typically, the thickness of the print wall is defined by the software through the line width specified. However, user can define a wall path when modelling for the object. This can be set in the slicing software if double, triple, or thicker walls are wanted.

2.6.2 Support

Support serves to hold up an overhang feature during printing. However, it is normally difficult to add the supported shape to the 3D clay model. For example, if a portrait is printed, the layers below the chin often hang too high so that the layers cannot come out. Hence, to overcome the problem, most modelling programs will make a narrow 45-degree nail under the chin that will be printed and then it can be cut when the printing is finished, and the clay hardens.

CHAPTER 3

METHODOLOGY AND WORK PLAN

3.1 Introduction

This section presents the methodology and work plan needed to be carried out to achieve the aim and objectives of this project. In this project, the material used for the printing was identified as lean clay. The clay was prepared with different water formulations to study the effect of moisture content on the printability of the clay. Then, the rheological properties of the clay samples were studied using a rheometer. After evaluating the rheological behaviours of the clays, various printing tests would be performed followed by sintering of the printed parts. In brief, the work plan of the project is being summarised in a flowchart as shown in Figure 3.1.

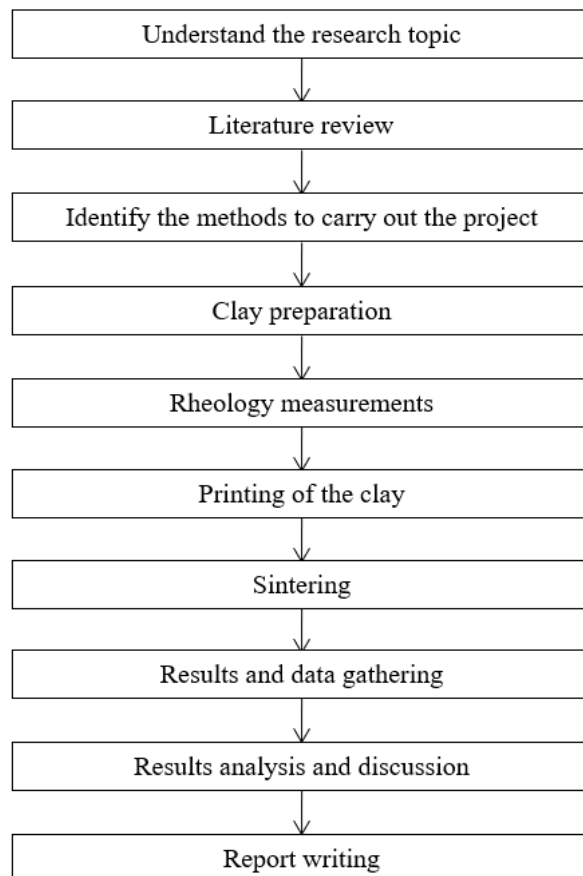


Figure 3.1: Work Flowchart.

3.2 Material

The material used in this project is a lean clay sample obtained from an oil and gas company named XSoil Lab Sdn Bhd. The Atterberg Limit test and particle size analysis have been carried out by XSoil Lab Sdn Bhd to investigate certain physical properties of the clay. Herein, Atterberg Limit test was performed to discover the plastic limit, liquid limit, and the plasticity index of the clay. Meanwhile, the particle size analysis was performed to measure the distribution of particle size in the clay. Figure 3.2 shows the raw clay sample obtained from the oil and gas company. A brief procedure for both the Atterberg Limit test and particle size analysis were presented as follows:

Atterberg Limit test (ASTM D4318, 2005):

Liquid Limit Procedure

- (i) Mix the clay until a consistency such that the number of drops required to close the groove are about 20 to 30.
- (ii) Place a portion of the clay into the liquid limit device as shown in Figure 3.3.
- (iii) Use the grooving tool to form a groove in the clay by drawing an arc against the surface of the liquid limit device, while keeping the tool in a perpendicular manner with the surface of the device.
- (iv) Turn the crank at a rate about 2 drops per second to lift and drop the device until the two separated clumps of clay encounter at the groove bottom.
- (v) Verify the closing of the groove at a distance of 13 mm.
- (vi) Record the number of drops needed for the groove to close.
- (vii) Add a small amount of clay to reform the specimen in the cup.
- (viii) Repeat steps 3 to 6 and ensure that the two successive tests have not more than two drops difference in the result.
- (ix) Determine the moisture content of the two specimens via the moisture content test and obtain the average of the results as the liquid limit of the clay.

Plastic Limit Procedure

- (i) Form a portion of clay in between 1.5 g to 2.0 g into an ellipsoidal mass.

- (ii) Use the plastic limit device as shown in Figure 3.4 to roll the clay into a thread with 3.2 mm diameter within 2 minutes.
- (iii) Break the thread into several pieces and reform these pieces into the ellipsoidal mass.
- (iv) Repeat the rolling, breaking and reforming processes the clay sample is not capable of being rolled into 3.2 mm diameter thread.
- (v) Collect the crumbled specimen in a container.
- (vi) Repeat steps 1 to 5 with the same clay sample.
- (vii) Determine the moisture content of the two specimens via the moisture content test and obtain the average of the results as the plastic limit of the clay.

Particle Size Analysis (ASTM D422, 2007):

- (i) Divide the portion of the clay onto the sieves with openings ranging from 0.075 mm to 6.300 mm.
- (ii) Perform the sieving process within 1 minute until the residue on the sieve is not more than 1% of the original mass.
- (iii) Determine the mass of each portion of the clay that passes the sieves and compute the percentage of clay passing the sieves.



Figure 3.2: Lean Clay Sample.



Figure 3.3: Liquid Limit Device.



Figure 3.4: Plastic Limit Rolling Device.

3.3 Determination of Moisture Content

A standard test method to determine the moisture content of the as-received lean clay was carried out. Herein, the test commenced by determining the mass of a dry and clean specimen container. After that, the moist test specimen would be placed in the container and the overall mass of the container and specimen would be measured. The container with the moist specimen was then placed in the drying oven as shown in Figure 3.5 until a constant mass is achieved. To obtain a constant mass of the specimen, the moist specimen was dried in the oven at a temperature of 110 ± 5 °C for around 24 hours. The mass of water is deemed to be the loss of mass after drying. In this test, the water content of the clay was calculated using the ratio of mass of water to the mass of dry specimen. The detailed calculation formula is shown as follows (ASTM-D-2216-98, 1998):

$$w = \left[\frac{(M_{cms} - M_{cds})}{(M_{cds} - M_c)} \right] \times 100 = \left(\frac{M_w}{M_s} \right) \times 100 \quad (3.1)$$

where

w = water content, %

M_{cms} = mass of container and moist specimen, g

M_{cds} = mass of container and oven dry specimen, g

M_c = mass of container, g

M_w = mass of water, g

M_s = mass of oven dry specimen, g



Figure 3.5: Drying Oven.

3.4 Preparation of the Paste Suspensions

The paste suspensions were prepared by adding distilled water into dry clay powder in varying amounts to obtain clay samples with different moisture content. Herein, the preparation of the suspension was conducted by manually mixing the clay powder and distilled water with spatula to acquire clay samples with water content ranging from 30% to 47%, as summarised in Table 3.1. Same clay powder was used to prepare all suspension with different water formulation; thus, the particles composition may be deemed to have minimal effect on the printed results (Revelo and Colorado, 2018). The amount of water that needs to be added in order to obtain the desired moisture content can be calculated via the following equation derived from equation 3.1:

$$M_w = \frac{w \times M_s}{100} \quad (3.2)$$

Table 3.1: The Formulation of the Clay Suspension for Different Moisture Content.

water content, w (%)	30	36	40	47
mass of dry clay, M_s (g)	250	250	250	250
mass of water to be added, M_w (g)	75	90	100	117.5

3.5 Evaluation of the Rheological Properties

The rheological properties of the clay samples with different water content were studied using a modular compact rheometer (MCR 301 Anton Paar Rheometer). To commence the rheology measurements, the rheometer was set up and initialised as shown in Figure 3.6. A parallel plate with 25 mm diameter was selected as the measuring system for the rheology tests, at 22 °C. The procedure was split into three parts, which are namely amplitude sweep, viscosity sweep, and frequency sweep to test different variables of the rheological properties. The detailed steps for each part were presented as follows:

Amplitude sweep:

- (i) Set an increasing shear strain from 0.0001% to 10%.
- (ii) Set a constant frequency at 1 Hz.
- (iii) Deposit the clay with moisture content of 47% on the Peltier plate of the rheometer.
- (iv) Move the parallel plate downward until a gap of 1 mm is achieved with the Peltier Plate.
- (v) Trim the excess clay from the sides of the plate.
- (vi) Run the test to determine the linear viscoelastic region of the clay from the graph of storage modulus vs shear stress.
- (vii) Take the plateau value in the linear viscoelastic region as the storage modulus of the clay sample.
- (viii) Find the drop in the shear stress from the graph plotted which indicates the static yield stress of the clay.
- (ix) Repeat the test for clay samples with moisture content of 40%, 36%, and 30%.

Viscosity sweep:

- (i) Set the shear rate range from 0.001 s^{-1} to 100 s^{-1} .
- (ii) Deposit the clay with moisture content of 47% on the Peltier plate of the rheometer.
- (iii) Move the parallel plate downward until a gap of 1 mm is achieved with the Peltier Plate.
- (iv) Trim the excess clay from the sides of the plate.
- (v) Run the test to obtain a graph of viscosity vs shear rate.
- (vi) Analyse the graph to investigate the viscosity behaviour of the clay at different shear rates.
- (vii) Repeat the test for clay samples with moisture content of 40%, 36%, and 30%.

Frequency sweep:

- (i) Set a constant shear strain at 0.001%.
- (ii) Set the frequency range from 1 Hz to 100 Hz.
- (iii) Deposit the clay with moisture content of 47% on the Peltier plate of the rheometer.
- (iv) Move the parallel plate downward until a gap of 1 mm is achieved with the Peltier Plate.
- (v) Trim the excess clay from the sides of the plate
- (vi) Run the test to get a graph storage modulus versus frequency.
- (vii) Analyse the graph to investigate the effect of frequency on the clay network and the storage modulus of the clay.
- (viii) Repeat the test for clay samples with moisture content of 40%, 36%, and 30%.



Figure 3.6: MCR 301 Anton Paar Rheometer.

3.6 3D Printing of the Clay Samples

The clay samples were printed by DIW technique using a Eazao Zero Printer. The printer used in this research is a screw-based extrusion printer which specialises in ceramic printing. Generally, the printer is made up of two components, which are the main body and the electric putter, as shown in Figure 3.7 (a) & (b). Herein, the main body serves as the printing base for the printer while the electric putter feeds the clay into the extruder for printing operation. The setup of the printer is presented in Figure 3.8. Two software which are namely Ultimate Cura and Clayon were used to assist in the G-code generation for the printing purposes. The clay samples with different water formulation were investigated through different printing tests as stated below:

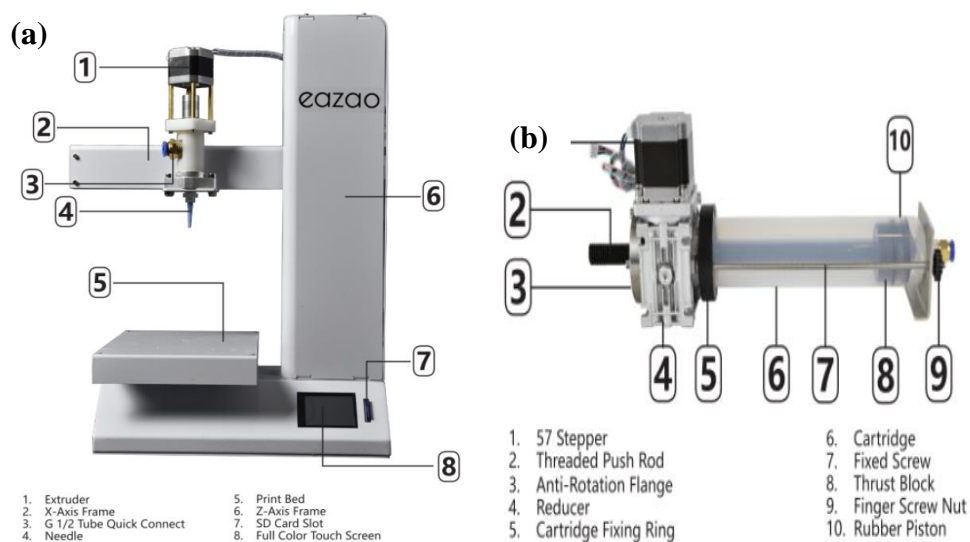


Figure 3.7: (a) Main Body of the Printer, (b) Electric Putter.



Figure 3.8: Setup of the 3D Printer.

3.6.1 Base Layer Tuning

In this test, the clay samples were tested at 47% moisture content through a nozzle diameter of 1.55 mm (14G). The base structure of the printed samples was a circle with 50 mm diameter. The base settings configuration was adjusted using Clayon software and the details were shown in Figure 3.9. Herein, two settings, which are base layer offset and number of base layers, were manipulated in this test. Base layer offset controls the distance between the outer edge of the base and the model while the number of base layers determines the quantity of base layers. Meanwhile, the base layer height and base layer ratio were kept constant at 1.0 mm and 50.0 respectively. The base layer height is a parameter that determines the height of the base layer, whereas the base layer ratio controls the clay amount being extruded when printing the base layer. The purpose of this test is to fine tune the base setting to obtain the best configuration for base printing.

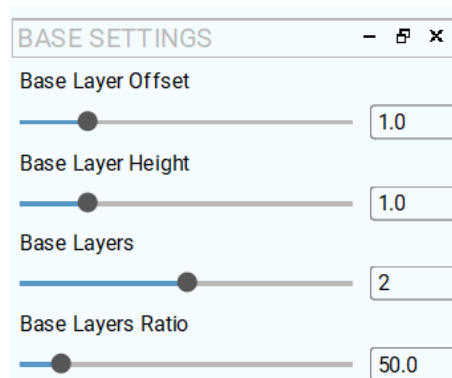


Figure 3.9: The Base Settings Configuration in Clayon Software.

3.6.2 Vertical Height Test

In this test, the clay samples were tested at 47% moisture content through nozzle diameter of 1.55 mm (14G) and 1.20 mm (16G). The test was conducted by allowing the clay samples to print vertically to the maximum sustainable height at different base diameters. In this context, four base diameters were investigated which are 20 mm, 30 mm, 40 mm, and 50 mm. The layer height and line width used were set equal to the nozzle diameter used during printing. A printing speed of 20 mm/s was used throughout this test. A sample configuration and sliced printing plan of the test is shown in Figure 3.10 (a) and (b). This test aims to investigate the relationship between the height and the base diameter of the clay samples through different nozzle sizes (layer height).

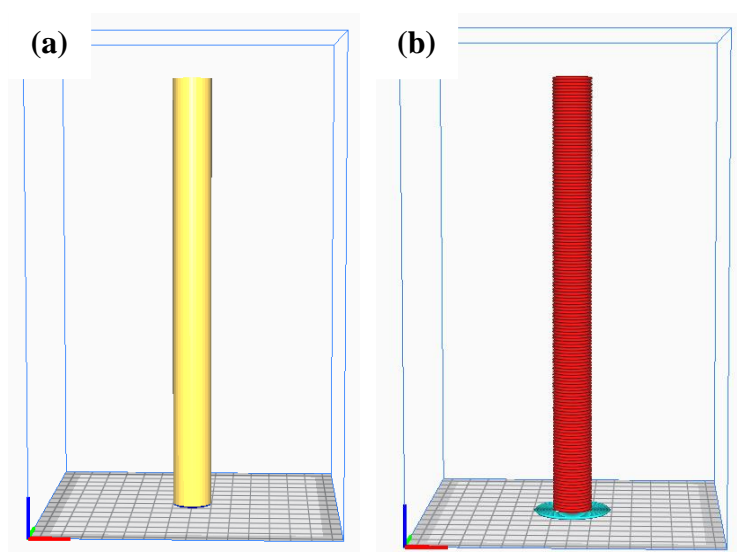


Figure 3.10: (a) Height Test Sample, (b) The Sliced Printing Plan for Height Test.

3.6.3 Nozzle to Layer Height Test

In this test, the clay samples were tested at different moisture content through various nozzle diameters ranging from 0.60 mm (20G) to 1.55 mm (14G). A vase with a diameter of 50 mm and height of 50 mm was designed to use as the sample configuration of the test. The test was conducted by printing the clay samples at different nozzle to layer height ratio. Herein, two ratios of 1:1 and 2:1 were used to evaluate the clay samples with different moisture content. For instance, when 1.20 mm nozzle diameter was selected, the layer height tested

would be 1.20 mm and 0.60 mm. A printing speed of 20 mm/s was used throughout this test. A sample configuration and sliced printing plan of the test is shown in Figure 3.11 (a) and (b). This test aims to study the effect of moisture content on the printability of the clay samples through different nozzle size (layer height).

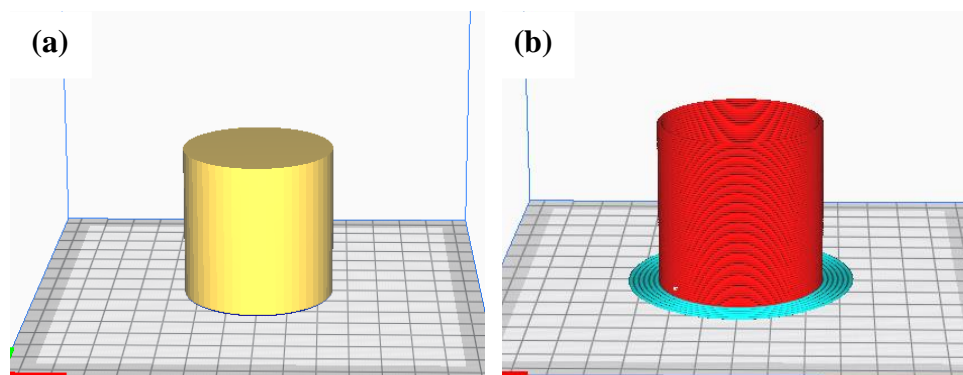


Figure 3.11: (a) Nozzle to Layer Height Test Sample, (b) The Sliced Printing Plan for the Test.

3.6.4 Angle Test

In this test, the clay samples were tested at various moisture content through nozzle diameter of 1.55 mm (14G). The layer height used for the test was half the nozzle diameter (0.775 mm). The test was conducted at 4 different angles of 30°, 45°, 60°, and 75°. The printing speed was maintained at 20 mm/s throughout the test. A sample configuration and sliced printing plan of the test is shown in Figure 3.12 (a) and (b). The purpose of this test is to find out the capability of the clays to sustain at different angles.

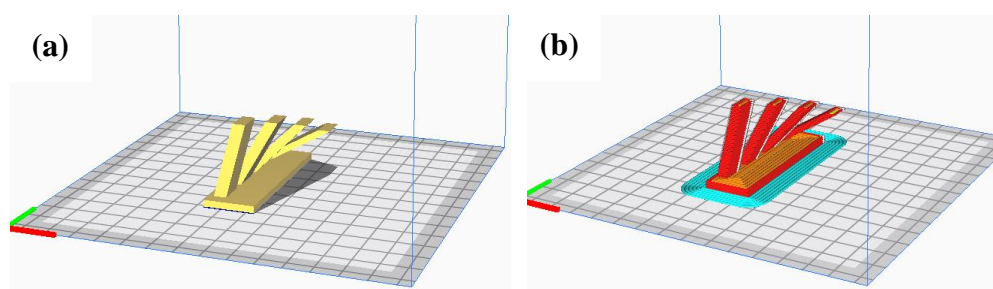


Figure 3.12: (a) Angle Test Sample, (b) The Sliced Printing Plan for Angle Test.

3.7 Sintering

After printing, the printed specimens were undergoing a drying process for a minimum of 24 hours by exposure to air at ambient conditions. Then, the dry specimens would be sintered in a heating furnace at 1000 °C for 30 minutes. During the heating, a temperature ramp of 5 °C per minute was applied, and the specimens were cooled naturally in air after sintering.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

In this chapter, the results obtained from the methodology as planned will be analysed and discussed. Herein, the characteristic of the lean clay sample will be evaluated. The rheological measurements will define the rheological behaviour of the clay samples at various water contents. Various printing tests have been performed to achieve the aim and objectives in this project. All the 3D printed specimens will be presented and analysed in detail. Finally, the sintered specimen will also be presented graphically for ease of comprehension.

4.2 Clay Characterisation

The results of the Atterberg Limit test are shown in Table 4.1. From the results, it denotes that the lean clay sample has a liquid limit of 47% and a plastic limit of 13%. The liquid limit indicates the moisture content at which the clay behaves at a boundary between the states of plastic and semi-liquid and plastic, whereas the plastic limit specifies the water content at which the clay changes from plastic state to semi-solid state (ASTM D4318, 2005). Eventually, the Atterberg limit test provides a good reference in determining the range of water content to be used in preparing the clay suspension.

Table 4.1: The Results of the Atterberg Limit Test.

Sample	Lean Clay
Plastic Limit (%)	13
Liquid Limit (%)	47
Plasticity Index (%)	34

Besides, Figure 4.1 displays the results for the particle size analysis of the raw lean clay sample. The results indicate that the sample consists of 26% clay, 51% silt, and 23% sand. The analysis also denotes the maximum particle diameter of 1.1800 mm. Herein, the sieve analysis further reveals that 98% of the particles in the sample are able to pass through the sieve opening of 0.600

mm. On the other hand, Table 4.2 displays the detailed result of the moisture content of the as-received clay sample. From the result obtained, the moisture content of the as-received clay sample was found to be 47%, which is the liquid limit of lean clay.

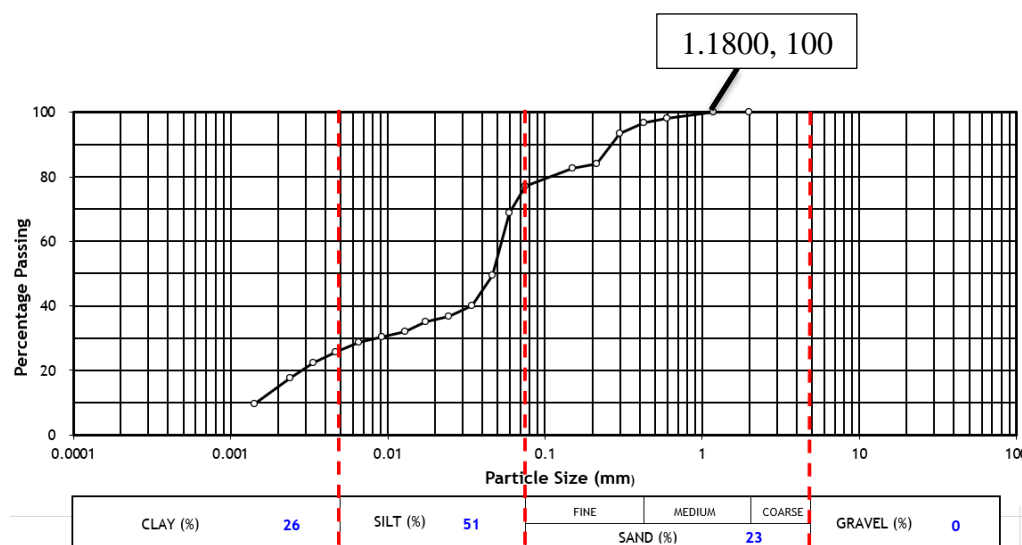


Figure 4.1: The Graph of Particle Size Analysis.

Table 4.2: The Results of Moisture Content Test for the As-Received Clay Sample.

Mass of container and moist specimen, M_{cms} (g)	138
Mass of container and oven dry specimen, M_{cds} (g)	106
Mass of container, M_c (g)	38
Mass of water, M_w (g)	32
Mass of oven dry specimen, M_s (g)	68
Moisture content (%)	47.06 \approx 47

4.3 Effect of Moisture Content on Rheology

Figure 4.2 shows the graph of viscosity versus shear rate for clay samples with moisture content ranging from 30% to 47%. From the graph, it can be observed that the viscosity exhibits a decreasing trend as the shear rate increases for all the clay samples. In other words, the viscosity decreases when the shear rate increases. Herein, the trend is generally caused by the intrinsic thixotropic behaviour which eventually result in the shear thinning property of the clay samples (Ordoñez, Gallego and Colorado, 2019). According to Chan *et al.*

(2020), the thixotropic behaviour is typical for the ink of DIW as it prevents the ink from dripping before printing and ensures continuous flow of the ink when adequate stress is applied. Besides, this characteristic also enables the clay to retain its shape after printing. In relation to the thixotropic behaviour, typical clay suspension will exhibit a shear thinning behaviour which can be represented by the Herschel-Bulkey model:

$$\tau = \tau_0 + k\dot{\gamma}^n \quad (4.1)$$

where

τ = shear stress, Pa

τ_0 = yield stress, Pa

k = consistency index, Pa·sⁿ

$\dot{\gamma}$ = shear rate, s⁻¹

n = flow index

Based on the Herschel-Bulkey model, a clay suspension will usually experience a progressive shear thinning process until the yielding of the clay (Barki, Bocquet and Stevenson, 2017). After the yielding, the shear thinning behaviour will stop and the subsequent structural regeneration will be experienced by the clay after the removal of shear load. This property will eventually ensure the printability of clay via DIW technique. When comparing between the four moisture content, it can be noticed that the viscosity of the clay decreases with the increase in water content. Similar trend can be observed in the research of Chan *et al.* (2020) which denotes the correlation in the rheology of the clay. In this context, it can be deduced that the lower the moisture content in the clay, the more viscous the suspension will become. As a result, the viscosity sweep provides a good insight in understanding the viscosity behaviour of the clay samples with different moisture content. Figure 4.3 and Table 4.3 summarise the shear thinning behaviour of the clay samples with different moisture content according to Herschel-Bulkey model.

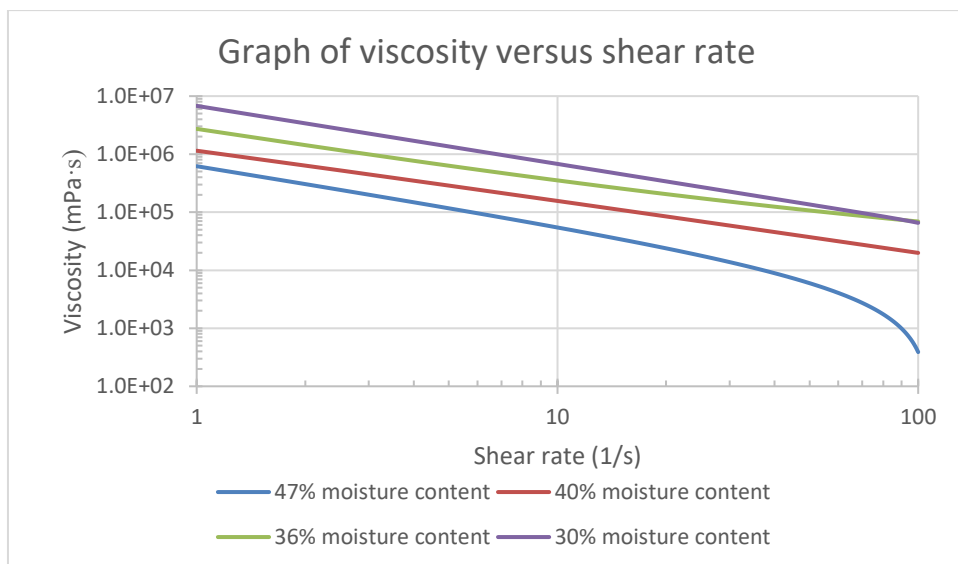


Figure 4.2: Graph of Viscosity Versus Shear Rate.

Table 4.3: The Shear Thinning Properties of the Four Clay Samples with Different Moisture Content

Moisture content (%)	Consistency index, k ($\text{Pa} \cdot \text{s}^n$)	Flow index, n
30	2235.12	0.1330
36	2065.38	0.2481
40	2014.65	0.2137
47	1346.48	0.3270

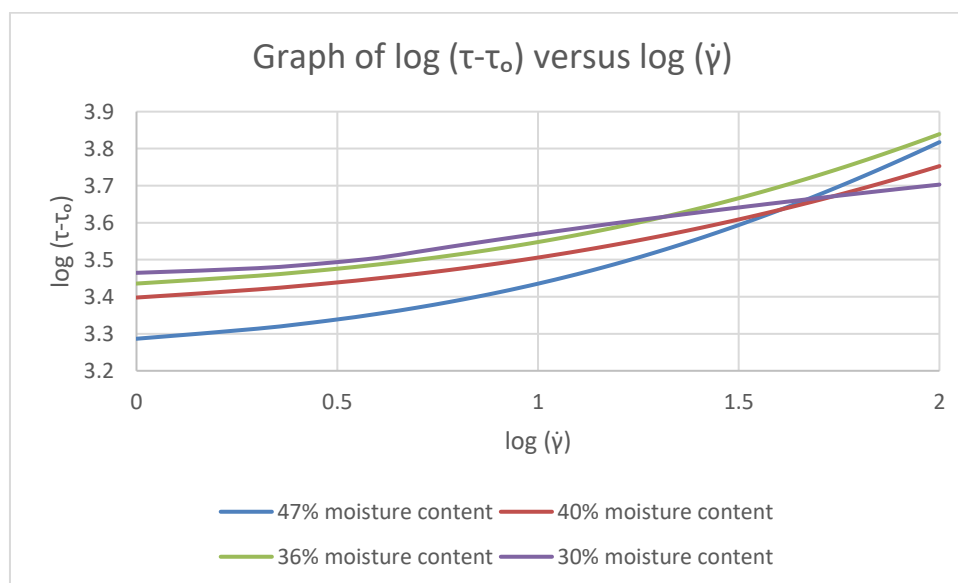


Figure 4.3: Graph of $\log(\tau - \tau_0)$ Versus $\log(\dot{\gamma})$.

In addition, Figure 4.4 shows the graph of storage modulus versus shear stress for clay samples with moisture content ranging from 30% to 47%. From the graph, it can be observed that the storage modulus plateau at the low shear stress region then begins to drop significantly when it reaches a certain threshold point for all four samples. Herein, the plateau of the storage modulus denotes the linear viscoelastic region of the clay, whereas the threshold point indicates the yield stress of the clay. From the graph, both storage modulus and yield stress of the clay samples can be extracted. Table 4.4 displays the storage modulus and yield stress of four clay samples with different moisture content. From the results extracted, it can be observed that the clay samples exhibit an increasing trend for both the storage modulus and yield stress as the water content decreases. These results correlate well with the research of Chan *et al.* (2020). In this project, the same clay powder was used to prepare all suspension with different water formulations. Hence, the particle characteristics such as size and shape of the particles may be deemed to have minimal effect on the rheological properties. In this context, it can be deduced that the water content affects the yield stress and storage modulus of the clay samples. According to Franks *et al.* (1999), when the water content decreases, the particles within a unit volume of the clay will increase, causing more interparticle bonds to be formed. As such, a higher stress is required to break the increasing interparticle bonds, thus resulting a higher yield stress and storage modulus.

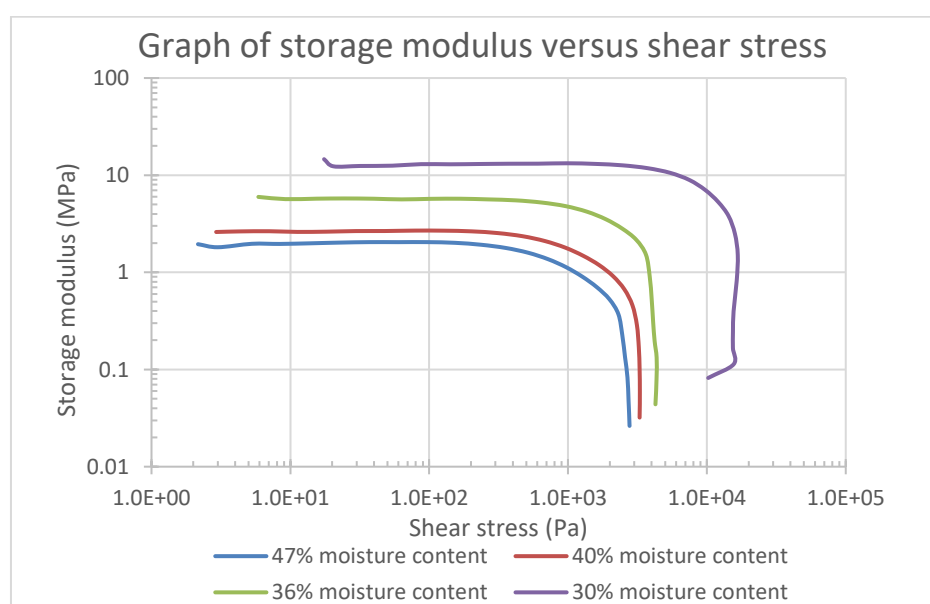


Figure 4.4: Graph of Storage Modulus Versus Shear Stress.

Table 4.4: The Storage Modulus and Yield Stress of the Four Clay Samples with Different Moisture Content.

Moisture content (%)	Storage modulus (MPa)	Yield stress (Pa)
30	13.14	2426.27
36	5.616	453.04
40	2.670	245.02
47	2.042	130.35

Moreover, Figure 4.5 displays the graph of storage modulus versus frequency for clay samples with moisture content ranging from 30% to 47%. From the graph, it can be observed that the storage moduli for all four clay samples are relatively constant as the frequency increases. This result denotes that the frequency possesses minimal effect on the storage modulus of the lean clay.

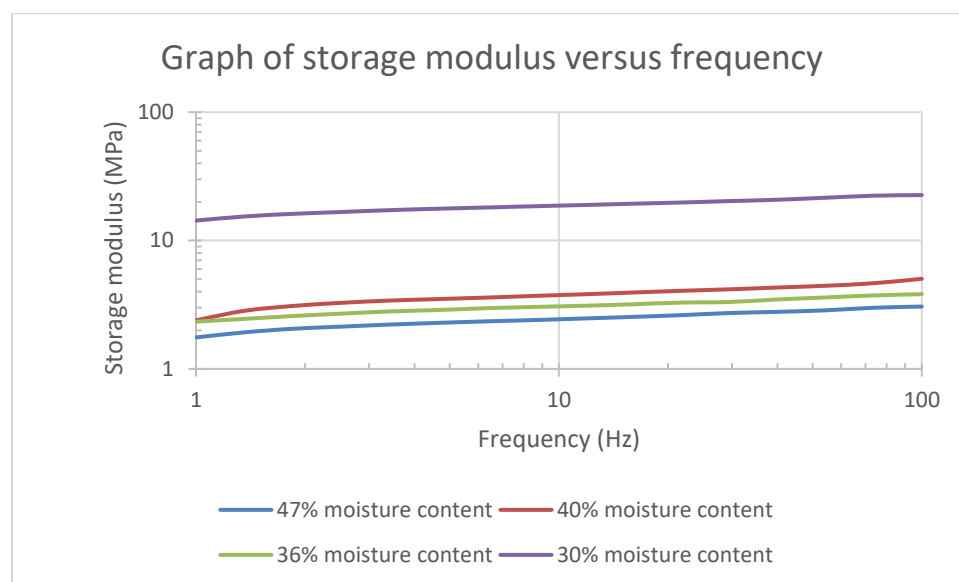
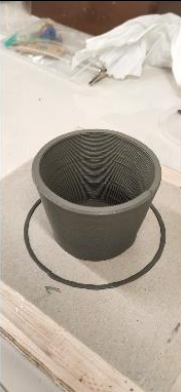

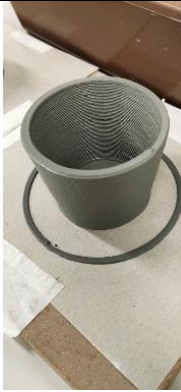



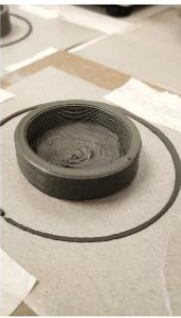

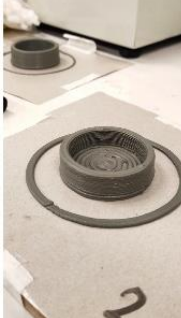





Figure 4.5: Graph of Storage Modulus Versus Frequency.

4.4 Results of Base Layer Tuning

In this test, the base layer offset and number of base layers were manipulated to investigate the effect of these settings on the quality of the base printed. Table 4.5 (a) and (b) summarise the results of the base tuning for before and after drying of the specimens.

Table 4.5: (a) The Results of Base Tuning Before Drying,

Number of layers	Base layer offset					
	1		3		5	
1						
2						

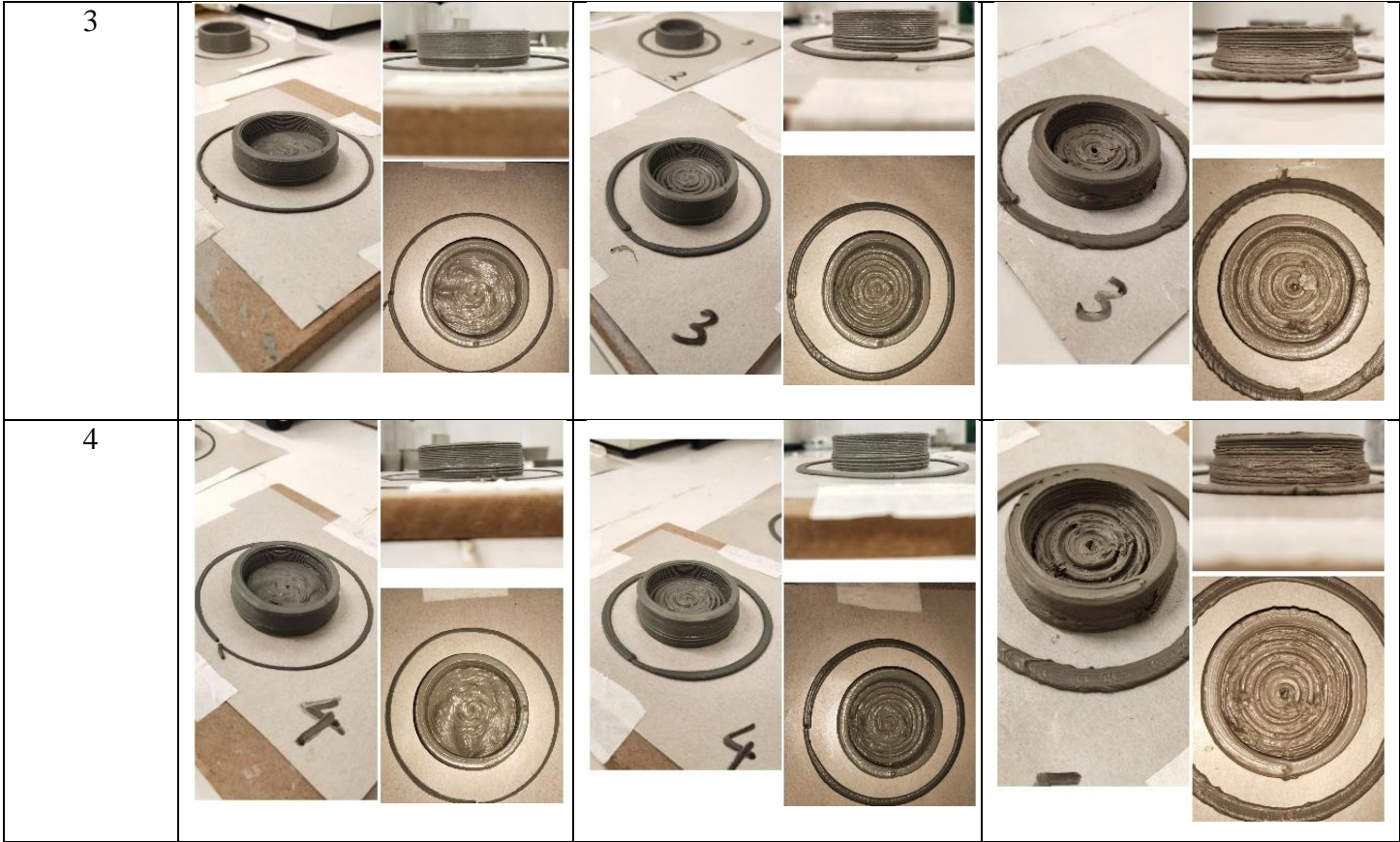
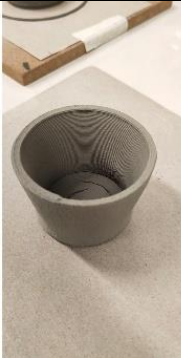











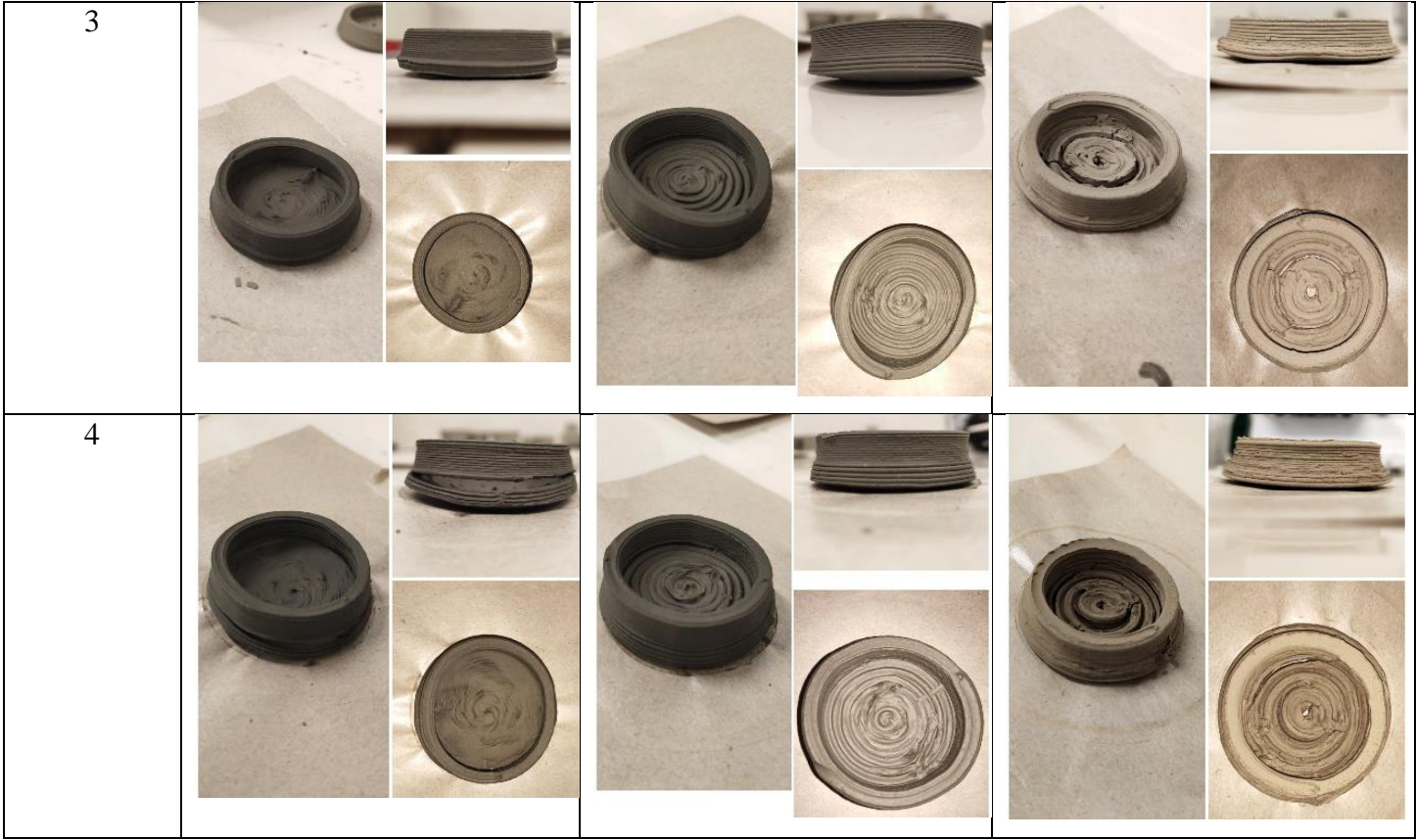


Table 4.5: (b) The Results of Base Tuning After Drying.

Number of layers	Base layer offset					
	1		3		5	
1						
2						



Based on the results shown above, it can be observed that as the base layer offset increases, the thicker the line width of the base becomes. In other words, the base layer offset affects the line width of the printed base. From the observation, the smaller the base layer offset, the closer the distance between two adjacent lines, thus the finer the base printed. Consequently, a smaller base layer offset will produce a higher quality base.

On the other hand, by comparing the printed specimens before and after drying, cracking of the base can be observed for all the specimens that have 2 base layers and less. The cracking occurs because the surface tension of the water exceeds the yield stress of the clay during drying. Herein, the specimens that have 3 or more base layers are deemed to be able to withstand the surface tension of the water during drying. As a result, the higher the number of base layers, the higher the strength of the base printed.

4.5 Effect of Moisture Content on Printability through Different Nozzle Diameters

In this part, the clay samples with moisture content ranging from 30% to 47% were evaluated for their printability through different nozzle sizes. In terms of the relationship between the moisture content and printability, it was discovered that the samples with moisture content that is below than 40% experience difficulty in printing. The suspensions are deemed to be too hard to be extruded by the electric putter of the printer, thus causing the clays to choke at the end of the putter. Hence, the moisture content of 40% can be deduced as the threshold value for the printability of the clay samples. Based on the rheological data obtained previously, it can be found that the clay sample with 40% water content possesses a storage modulus of 2.670×10^6 Pa. Below this threshold value of storage modulus, printing is deemed to be feasible. Table 4.6 summarises the printability of the clay samples at different moisture content.

Table 4.6: Printability of the Clay Samples at Different Moisture Content.

Moisture content (%)	Printability
30	Not printable
36	Not printable
40	Printable
47	Printable

In terms of the printability of the clay samples through different nozzle sizes, it was found that below the nozzle diameter of 1.20 mm (16G), the suspensions will experience clogging at the nozzle, preventing the ink to be extruded for printing. Herein, it is presumed that the clogging occurs due to the blocking of large particles at the nozzle during printing (Chan *et al*, 2020). Based on the particle size analysis discussed above, it was discovered that the lean clay sample possesses the maximum particle diameter of 1.1800 mm. Hence, in order for the clay to be printed, a nozzle diameter that is greater than 1.1800 mm should be used. Table 4.7 shows the printability of the clay samples at different nozzle sizes.

Table 4.7: Printability of the Clay Samples through Different Nozzle Diameters.

Nozzle diameter (mm)	Printability	
	40% wt	47% wt
0.60 (20G)	Not printable	Not printable
0.84 (18G)	Not printable	Not printable
1.20 (16G)	Printable	Printable
1.55 (14G)	Printable	Printable

4.6 Results of Nozzle to Layer Height Test

This part seeks to investigate the relationship between nozzle size and layer height in the clay printing. Besides, the results also serve as the proof for the printability of the clay samples with different moisture content through various nozzle sizes. Table 4.8 (a) and (b) summarises the results of nozzle to layer height test for the clay samples with 40% and 47% moisture content.

Table 4.8: (a) The Results of Nozzle to Layer Height for Clay Sample with 40% Moisture Content,

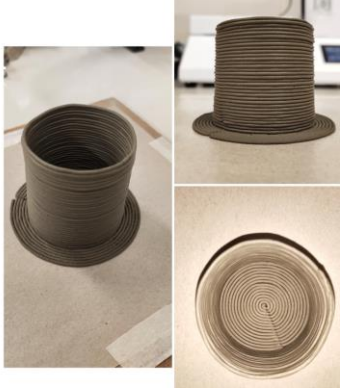

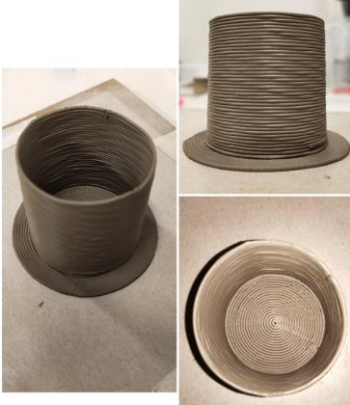

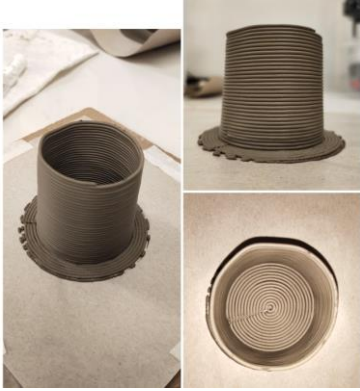

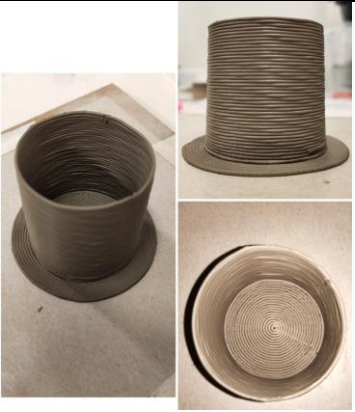

Moisture content (%)	40	
Nozzle : Layer Height	1:1	2:1
D-1.55 mm (14G nozzle)		
Layer height	1.55 mm	0.775 mm
D-1.20 mm (16G nozzle)		
Layer height	1.20 mm	0.60 mm

Table 4.8: (b) The Results of Nozzle to Layer Height for Clay Sample with 47% Moisture Content.

Moisture content (%)	47	
Nozzle : Layer Height	1:1	2:1
D-1.55 mm (14G nozzle)		
Layer height	1.55 mm	0.775 mm
D-1.20 mm (16G nozzle)		
Layer height	1.20 mm	0.60 mm

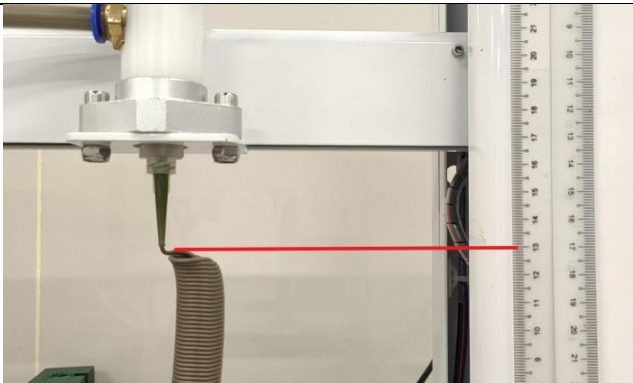
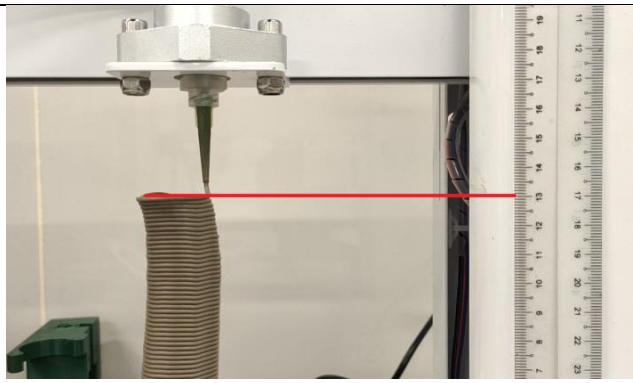
Based on the previous discussion in 4.5, it was found that the clays with moisture content of 40% to 47% produce printable results. In addition, the nozzle diameters in the range of 1.20 mm to 1.55 mm were able to produce printing without clogging. Herein, the results above correspond with the previous discussion. In terms of the relationship between the nozzle size and layer height, the results have shown that the ratio of 1:1 and 2:1 nozzle diameter to layer height are deemed to be the workable parameters for printing (Keep, 2020). A further observation is that the smaller the layer height, the finer the

wall quality of the specimen. In contrast, the nozzle diameter appears to have minimal effect on the wall quality printed.

4.7 Results of Height Test

In this test, the clay samples were printed to maximum sustainable height at various base diameters through different nozzle sizes. The moisture content of the clay is kept constant at 47% in this test. Table 4.9 (a) and (b) show the results of the height test for nozzle diameter of 1.55 mm (14G) and 1.20 mm (16G) respectively.

Table 4.9: (a) The Results of Height Test for Nozzle Diameter of 1.55 mm,

Nozzle diameter	1.55 mm (14G)	
Diameter (mm)	Height (mm)	L/D ratio
20	 <p style="text-align: center;">$Z \approx 130$ mm</p>	6.5
30	 <p style="text-align: center;">$Z \approx 130$ mm</p>	4.33

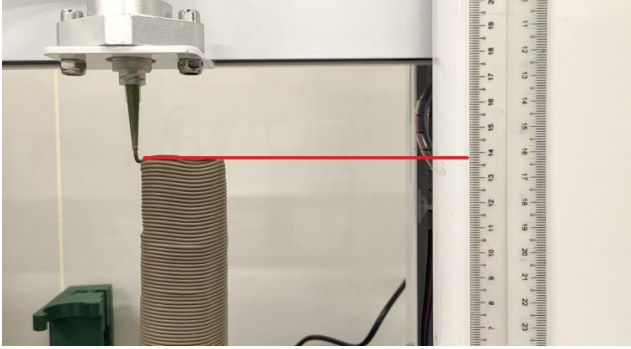
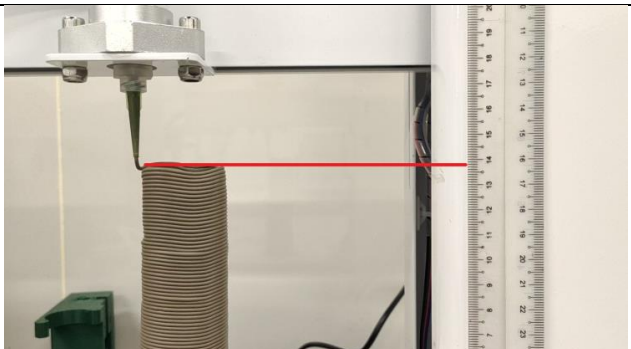
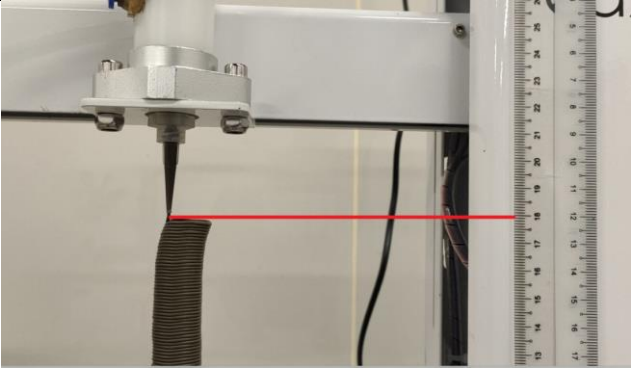
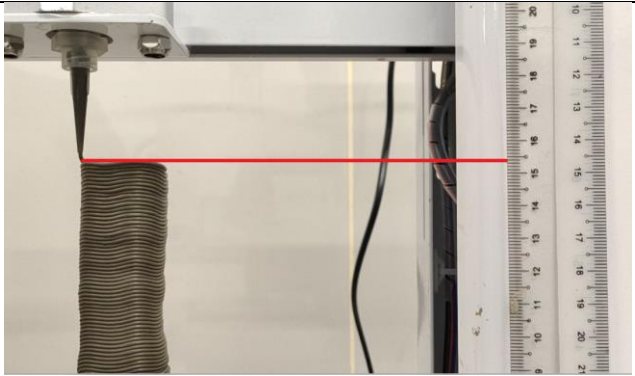
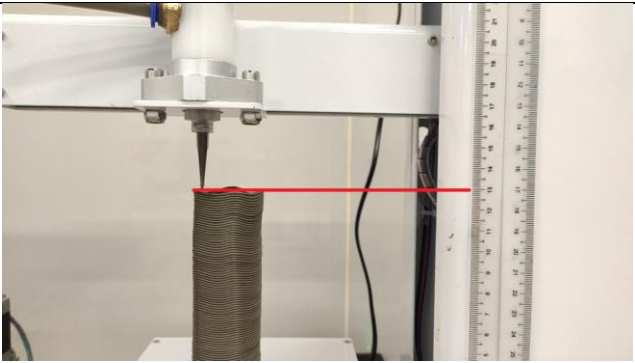
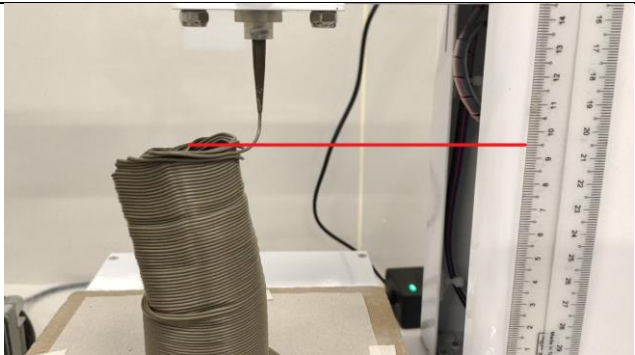
40	 <p style="text-align: center;">$Z \approx 138 \text{ mm}$</p>	3.45
50	 <p style="text-align: center;">$Z \approx 115 \text{ mm}$</p>	2.30

Table 4.9: (b) The Results of Height Test for Nozzle Diameter of 1.20 mm.

Nozzle diameter	1.20 mm (16G)	
Diameter (mm)	Height (mm)	L/D ratio
20	 <p style="text-align: center;">$Z \approx 180 \text{ mm}$</p>	9.00

30	 <p style="text-align: center;">$Z \approx 154 \text{ mm}$</p>	5.13
40	 <p style="text-align: center;">$Z \approx 130 \text{ mm}$</p>	3.25
50	 <p style="text-align: center;">$Z \approx 95 \text{ mm}$</p>	1.90

From the results obtained, a graph of L/D versus base diameter is plotted, as shown in Figure 4.6. From the graph plotted, it can be observed that the L/D ratio for both nozzle diameters experience a decreasing trend as the base diameter increases. In other words, the maximum sustainable height of the clay decreases as the nozzle size increases. When comparing between two nozzle diameters, it is noticed that at small base diameter (20 mm and 30 mm), the L/D ratio of the printed results for nozzle diameter of 1.20 mm are relatively higher than that of 1.55 mm nozzle diameter. As the base diameter increases, the L/D ratio of 1.55 mm nozzle diameter then becomes slightly larger than 1.20 mm

nozzle diameter. Herein, it can be postulated that a smaller nozzle diameter produces a finer wall, thus a stronger structure. In terms of rheology, a smaller nozzle diameter will eventually result in a thinner line being printed. In this context, a thinner line eventually yields a larger surface area, thus allowing more interparticle bonds to be formed (Chan *et al*, 2020). With increasing interparticle bonds, the attraction force between particles will become stronger; hence resulting in a stronger structure.

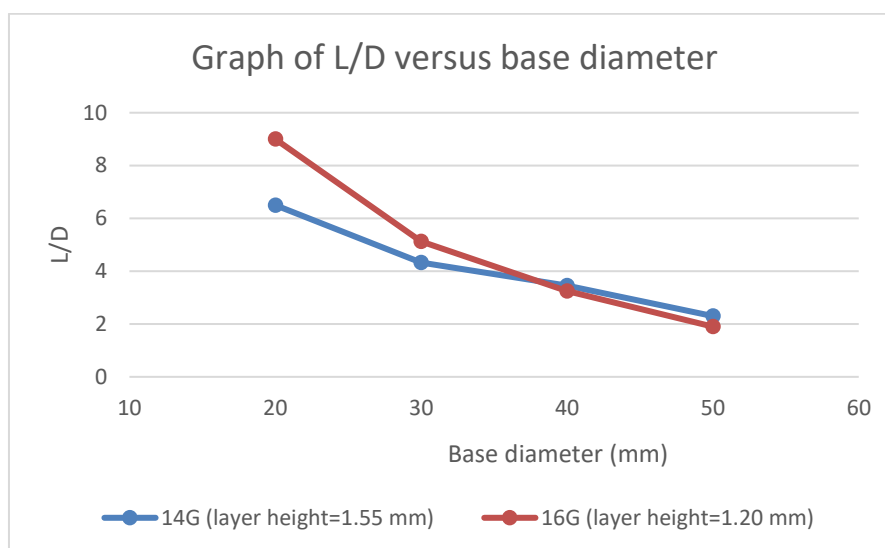
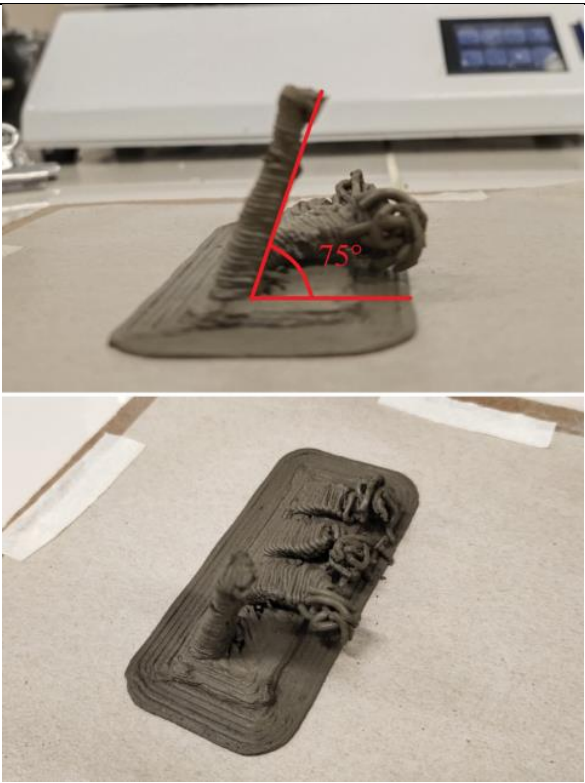
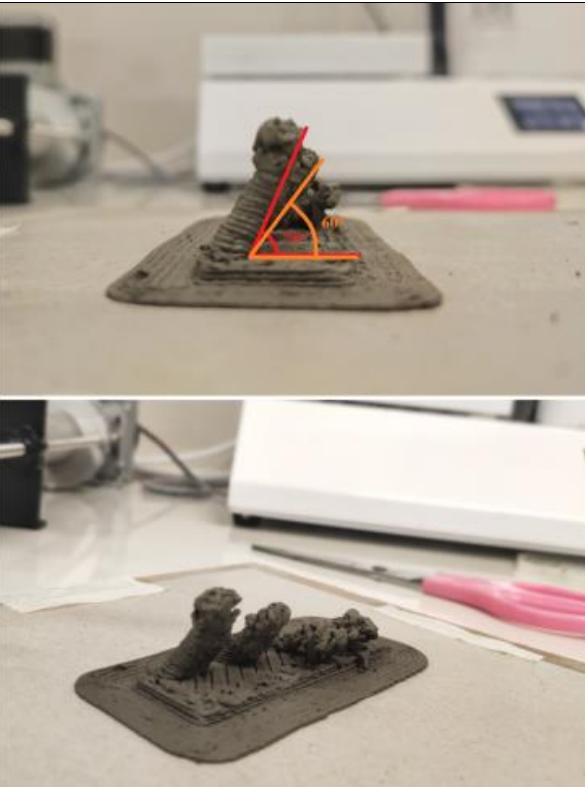


Figure 4.6: Graph of L/D Versus Base Diameter.

4.8 Results of Angle Test

In this test, the clay samples with moisture content of 40% and 47% were printed at various angles to assess the capability of the clays in sustaining different angles. The nozzle diameter is kept constant at 1.55 mm and the layer height used is 0.775 mm. Table 4.10 shows the results of angle test for both clay samples with moisture content of 47% and 40%.

Table 4.10: The Results of Angle Test for Clay Samples with Moisture Content of 47% and 40%.

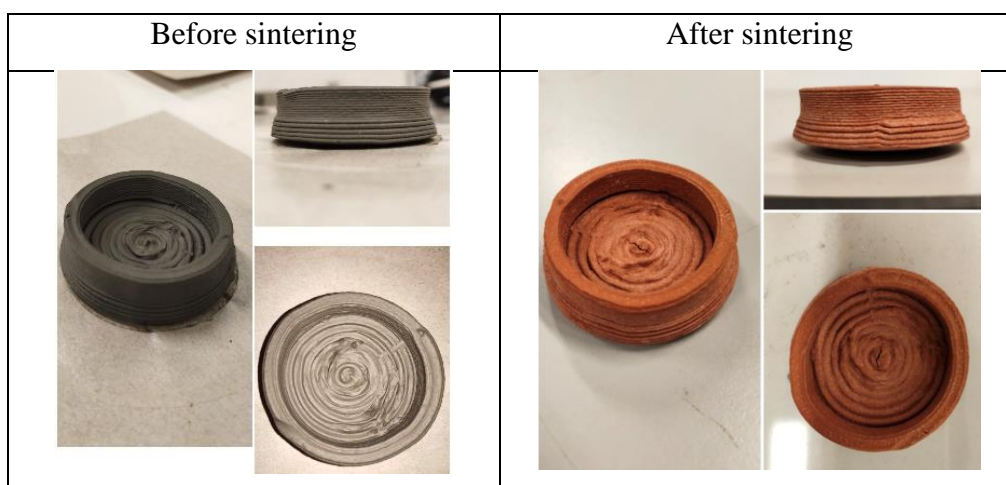
Moisture content (%)	Observation
47	
40	

From the results obtained, it can be observed that 75° is the sole angle which can be sustained by the clay with moisture content of 47%. For the angles of 60° , 45° , and 30° the structures collapsed during printing. Meanwhile, the clay with 40% moisture content is capable of sustaining both the angles of 75° and 60° . As a result, it can be postulated that the lower the water content in the clay, the higher its capability in sustaining different angles. In this context, the previous discussion has mentioned that the clay sample with lower water content yields a higher storage modulus. With a higher modulus, the interparticle bonds become harder to be broken; hence resulting in a higher structural integrity (Franks *et al*, 1999). Consequently, it can be deduced that the clay with lower moisture content possesses higher strength, making it able to withstand more inclined angle.

4.9 Sintered Results

Table 4.11 shows a printed specimen before and after it has undergone the sintering process. From the result obtained, a noticeable colour change of the specimen from grey to brown can be observed after undergoing sintering.

Table 4.11: Printed Specimen Before and After Sintering.



CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In conclusion, the main aim and objectives of this project have been fulfilled after carrying out the methodology and work plan. In this project, the moisture content of the lean clay was varied ranging from 30% to 47% and the rheological properties of each formulation were studied. Then, the rheological behaviours were correlated with the printability to study the printability criterion of lean clay. Herein, three different tests which are namely viscosity sweep, amplitude sweep, and frequency sweep were performed to gain a better insight of the rheological properties of the clay with different moisture content. From the result of viscosity sweep, it can be deduced that the viscosity of the clay decreases as the shear rate increases regardless of the moisture content. This property ensures the printability of lean clay using DIW technique. The amplitude sweep then provides the results of yield stress and storage modulus of the clay samples at different moisture content. An increasing trend can be obtained for both the storage modulus and yield stress as the water content decreases. Moreover, the frequency sweep denotes that the frequency does not possess a significant effect on the storage modulus of the clay samples.

After understanding the rheological properties of lean clay, the effect of the rheology on printability through different nozzle diameters was evaluated. Herein, the clay samples with moisture content between 40% to 47% are deemed to be printable. In contrast, the formulations with moisture contents below than 40% experience difficulty in printing. From the results obtained, it can be deduced that the moisture content of 40% is the threshold value for the printability of the clay samples. Meanwhile, in terms of the printability of the clay samples through different nozzle sizes, it was found that clogging is deemed to have occurred for the nozzle diameter below 1.20 mm (16G). Herein, it was postulated that the printability through different nozzle sizes is independent of rheological properties. Instead, it might be related to the particle

size of the clay samples. When the nozzle diameter exceeds the maximum particle diameter of the clay samples, printing is deemed to be feasible.

Besides, various printing tests have been performed to access the optimal printing criterion of lean clay. From the results of base tuning, it is deduced that a smaller base layer offset produces a higher quality base; higher number of base layers yields greater strength base. In addition, the results of the height test indicate that the L/D ratio of the printed specimen experiences a decreasing trend as the base diameter increases. Moreover, it is also deduced that a smaller nozzle diameter produces a finer wall, thus a stronger structure. Furthermore, the results of angle tests indicate that the lower the water content in the clay, the higher its capability in sustaining different angles. In other words, it can be deduced that the clay with lower moisture content possesses higher strength, making it able to withstand more inclined angle.

5.2 Recommendations for future work

Future work can be done by mixing the clay using moisture content at a smaller interval. In this project, there are four moisture content of 30%, 36%, 40%, and 47% being tested. From the results obtained, it was found that the moisture content ranging from 40% to 47% produces the clay samples that are printable. Hence, a more detailed distribution of the water content can be tested within the range of 40% to 47%. This will eventually enhance the understanding of the relationship between the water content, rheological properties, and the printability of the lean clay sample.

Besides, a wider range of nozzle diameters can be tested in the future to gain better insight on the printability of lean clay through different nozzle sizes. In this project, the nozzle diameters being tested are ranging from 0.60 mm to 1.55 mm. Herein, clogging is deemed to have occurred at the nozzle diameter below 1.20 mm due to the blocking of large particles at the nozzle during printing. Hence, more nozzle size should be adopted in future work to prove the presumption that the printability through various nozzle diameter is independent of rheology of the clay.

Due to the time constraint, the effect of sintering on the physical properties of the clay samples is not studied in this project. Hence, future work

should investigate the physical properties such as density and strength of the clay samples after sintering to have a better understanding on how the sintering process alters the physical properties of lean clay.

Finally, a different type of clay can be studied in the future to explore more material to be used in the 3D printing of ceramics. By exploring more material, the versatility of the raw material in ceramic printing can eventually be enhanced.

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APPENDICES

Appendix A: Results of Sieve Analysis

Sampel No. :	0		
Depth (m) :	0.0		
Wt of Dry Sample, (g)	50.0		
<u>SIEVE ANALYSIS</u>			
Sieve Opening (mm)	Weight Retained (g)	Percent Retained (%)	Percent Finer (%)
6.300	0.00	0.00	100
4.750	0.00	0.00	100
2.360	0.00	0.00	100
2.000	0.00	0.00	100
1.180	0.00	0.00	100
0.600	0.89	1.78	98
0.425	0.68	1.36	97
0.300	1.66	3.32	94
0.212	4.69	9.38	84
0.150	0.76	1.52	83
0.075	2.83	5.66	77

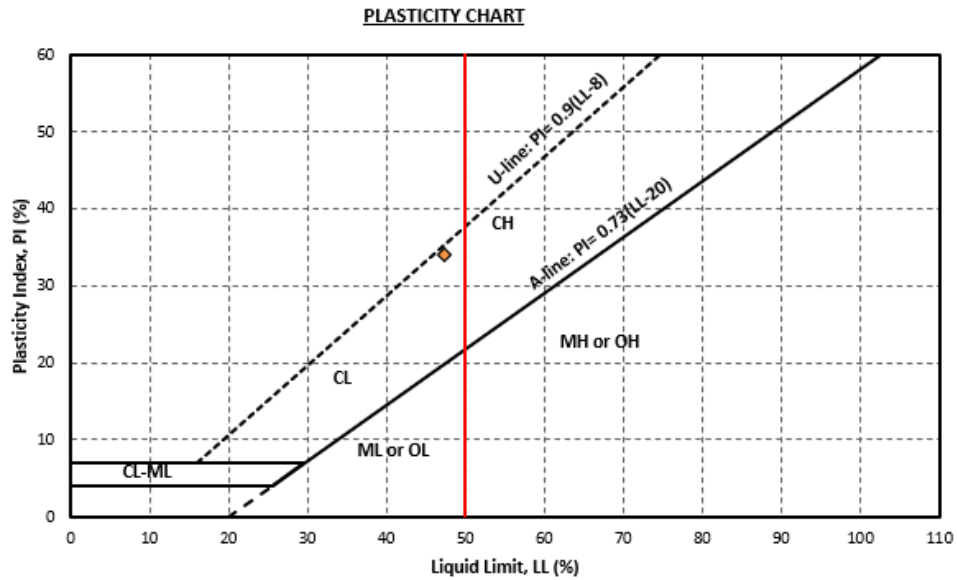
Appendix B: Test Report of Moisture Content for the As-Received Clay
Sample

TEST REPORT

DETERMINATION OF MOISTURE CONTENT (ASTM D2216-19)

Sample No.	KSW-DC-99			
Depth (m)	0.60-0.80			
Tare No.	XSL117	A92		
Wt. of Wet Soil + Tare (g)	92.23	82.89		
Wt. of Dry Soil + Tare (g)	71.16	64.04		
Wt. of Tare (g)	12.74	11.95		
Wt. of Water (g)	21.07	18.85		
Wt. of Dry Soil (g)	58.42	52.09		
Moisture Content (%)	36.1	36.2		
Average Moisture (%)	36			
Sample No.				
Depth (m)				
Tare No.				
Wt. of Wet Soil + Tare (g)				
Wt. of Dry Soil + Tare (g)				
Wt. of Tare (g)				
Wt. of Water (g)				
Wt. of Dry Soil (g)				
Moisture Content (%)				
Average Moisture (%)				

Appendix C: Plasticity Chart of Lean Clay



ML : Inorganic silts of low plasticity
 CL : Inorganic clays of low to medium plasticity
 OL : Organic silts of low plasticity
 MH : Inorganic silts of high plasticity
 CH : Inorganic clays of high plasticity
 OH : Organic silts of medium to high plasticity

Sample No.	Depth (m)	LL	PL	PI	Fines passing No.200 sieve	Soil Type
◆ KSW-DC-99	0.60-0.80	47	13	34	77	Lean CLAY, with sand

Appendix D: Test Report of the Atterberg Limit Test

TEST REPORT**LIQUID LIMIT, PLASTIC LIMIT & PLASTICITY INDEX (ASTM D4318-17)**

LIQUID LIMIT (LL) - ONE POINT METHOD B						
Test No.	1	2	1	2	1	2
No. of Blows	26					
Tare No.	XSL58	XSL47				
Wt. of Wet Soil + Tare (g)	24.56	27.49				
Wt. of Dry Soil + Tare (g)	18.81	21.15				
Wt. of Tare (g)	6.65	7.60				
Wt. of Dry Soil (g)	12.16	13.55				
Moisture Content (%)	47.3	46.8				

PLASTIC LIMIT (PL)						
Tare No.	A42	A20				
Wt. of Wet Soil + Tare (g)	24.67	23.49				
Wt. of Dry Soil + Tare (g)	22.69	21.73				
Wt. of Tare (g)	7.89	8.14				
Wt. of Dry Soil (g)	14.80	13.59				
Moisture Content (%)	13.4	13.0				

SUMMARY			
Sample No.	KSW-DC-99		
Depth (m)	0.60-0.80		
Liquid Limit (LL) (%)	47		
Plastic Limit (PL) (%)	13		
Plasticity Index (PI) (%)	34		
Soil Description	Brown (10YR 6/3) Lean CLAY, with sand		