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Thixotropy Evaluation on 3D Printing of Clay

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Abstract

3D printing of clay is a new additive manufacturing (AM) technique that has emerged as a technology for manufacturing shapes with complex geometries like Fused Deposition Modelling (FDM) polymer 3D printing. The goal of this study is to evaluate the flowability and buildability of ceramic clay that is continuously extruded by varying printing speed, nozzle diameter and layer height to determine how the process parameters affect the quality of the ceramic clay layering and printing. The fundamental component of this research effort is Sayong clay which can only be found in Kuala Kangsar, Malaysia. The results showed that Sayong clay has been successfully 3D printed, which has its characteristics with optimized parameters. A density test was performed, and the results reveal that density decreases as the water content rises. As the diameter of the nozzle decreases, the diameter of the wall extruded from the nozzle decreases as well. Thus, the wall diameter will expand less after printing and the precision of the printing will be more accurate to the actual model. The layer height test shows that the thinner the layer height, the finer the quality of print which leads to lower yield of defects and fractures to the products.

Keywords
Additive Manufacturing, Clay 3D Printing, Ceramic, Sayong Clay, Thixotropy

1. Introduction
Additive manufacturing (AM) converts stereolithography (STL) files derived from computer-aided design (CAD) models into layered instructions for fabrication (Wong & Hernandez, 2012). This process involves approximating CAD models with triangular meshes and slicing them into layers, each containing specific data for the final object. Fused deposition modelling (FDM), a widely utilized AM technique, fabricates objects by sequentially depositing layers of melted thermoplastic filament. The mechanical properties of FDM-produced components are influenced by variables such as material composition, extrusion temperature, printing parameters, and environmental conditions.
Extrusion-based ceramic 3D printing broadens the applications of AM by enabling the production of ceramic components.

This technique begins with ceramic clay loaded into a pressurized storage silo, transferred to the nozzle via a tube, and extruded using a screw mechanism driven by a stepper motor. The extruded clay filaments are deposited layer by layer according to CAD instructions, and the final ceramic structure is obtained through drying and sintering. To ensure effective ceramic 3D printing, continuous material delivery to the nozzle, precise filament diameter regulation, and accurate control of deposition trajectories are essential for layer consistency. Additionally, maintaining substrate temperature is critical for optimizing clay deposition and solidification processes. Real-time monitoring of extrusion and deposition allows for parameter adjustments, enhancing accuracy and overall print quality. These capabilities make ceramic 3D printing a precise and innovative approach to manufacturing complex ceramic structures.
Ceramic 3D printing technology faces significant challenges, including issues related to fractures, distortion, suboptimal mechanical properties, low yield, and limited accuracy of the manufactured components. The forming techniques utilized in clay-based 3D printing vary depending on material properties, environmental conditions, and

the power of the printing machinery, necessitating improvements in forming processes to enhance performance (Chao & Liu, 2019). Unlike commonly used materials such as polymers and metals, clay requires additional time to harden and develop adequate flexibility to support the weight of successive layers during the printing process (Wi et al., 2020).

The preparation of clay for 3D printing involves softening base clay powder with water and ensuring complete hydration by submerging the mixing clay, allowing water to penetrate even the smallest particles. To achieve the desired flexibility and hardness, the clay must be allowed sufficient time to dry before use. Notably, freshly mixed clay derived from dry powder often has a grainy texture and lacks plasticity, attributes that can influence print quality. Proper preparation and handling of clay are therefore critical to optimizing the performance and outcomes of ceramic 3D printing (Keep, 2020).

1.1 Objectives

The objectives of this research project are to measure the flowability and buildability of continuous extrusion of ceramic clay by varying the process parameter. Thus, to analyse the influence of the process parameters on the ceramic clay layering quality and defects of printed parts. The effects of nozzle geometries on 3D Printing of Clay also will be accounted as the objectives the research. The diameter of the nozzle was utilised between the sizes of 1.25 mm, 1.75 mm and 2.15 mm. Variables such as layer height were used when it came to buildability, although the nozzle shape was set to 0.5 mm, 1.0 mm and 1.5 mm. A basic shape that consists of various features with a dimension of 110 mm length, 110 mm width and 30 mm height.

2. Literature Review

Traditional ceramic forming methods, such as injection moulding, die pressing, tape casting, and gel casting, involve shaping a mixture of powder with or without binders and additives. These methods require sintering green parts at high temperatures to achieve densification. However, they are limited by long production times, high costs, and challenges in machining due to ceramics' brittleness. Defects such as cracking, poor surface quality, and dimensional inaccuracies further complicate the process. The integration of 3D printing into ceramic manufacturing addresses many of these issues. Since its initial exploration in the 1990s by Marcus et al. and Sachs et al., advancements in materials and computer science have expanded 3D printing technologies for ceramics. These technologies are categorized based on feedstock type: slurry-based, powder-based, or bulk solid-based. This study focuses on bulk solid-based approaches, including Fused Deposition Modelling (FDM) and Paste Deposition Modelling (PDM), which offer promising solutions for efficient and precise ceramic manufacturing (Chen et al., 2019).

In Fused Deposition Modelling (FDM), a filament is melted and extruded through a nozzle, hardening upon deposition and cooling. Paste Deposition Modelling (PDM) similarly extrudes paste at room temperature, solidifying through solvent evaporation (Ruscitti et al., 2020). FDM technology in ceramics offers advantages over traditional methods, including reduced production time, lower costs, and expanded design possibilities, particularly for low-volume or custom orders. The ISO standard categorizes clay 3D printers based on extruder function, operating mechanisms, and power requirements. Extrusion systems can be single-stage, utilizing pistons or screws, or two-stage, involving material transfer via pneumatic or mechanical systems for enhanced efficiency.

Thixotropy refers to the reversible transformation of a material from a fluid state to an elastic, solid-like gel. Initially introduced by Peterfi (1927) and Freundlich (1935), the concept is widely applied in geology and soil mechanics. Mitchell (1961) described thixotropy as an isothermal, time-dependent process where materials stiffen at rest but soften upon agitation or remoulding. ASTM (2014) defines it as the ability of a material to stiffen quickly when stationary and revert to a fluid state upon manipulation. Thixotropic behaviour arises from the reversible destruction and repair of molecular structures in the material. Its effects vary with the type of clay minerals, with water-absorbing minerals exhibiting stronger thixotropic properties. This characteristic benefits construction by increasing soil strength over time, enhancing structural safety. However, challenges such as material handling and temporary soil strength loss during pile driving exist. Thixotropic fluids, like drilling muds, are also critical in drilling applications.

3. Methods

Sayong clay obtained from a site visit in Kuala Kangsar, Perak was chosen to test its optimum layering quality and low defects. This method began with the selection of a clay material suitable for 3D printing, followed by the mixing of the clay with water and testing flowability. A 3D printer will extrude the clay mixture, and the resulting part will be examined for buildability. To draw conclusions and make recommendations for future research, the findings and analysis are documented.

3.1 Materials

The clay mixture materials selected for ceramic clay 3D printing have included local clay such as Sayong clay and Coldstream clay. Bentonite powder and water will act as their binder agent. Sayong clay is a component of the clay mixture that can be obtained in Kuala Kangsar, Perak. Sayong clay is unique because of the numerous raw material sources available in the Sayong village near Kuala Kangsar, Perak. The local entrepreneur uses this sand because of its unique ability to build up and maintain the position and shape of the products. Another local clay is Coldstream clay, which originated in the city of Bidor in Perak. The similarities between the components of this material and those of traditional porcelain, it can serve as an alternate body for porcelain. Bentonite is a very old clay that has been utilized as a cure for a variety of ailments. Bentonite is known as a swelling clay because of its ability to absorb large amounts of water. Clean water is a crucial component in the manufacture of clay mixtures. The first hydration process of clay for the binder is started with water. Platelets break apart and disperse because of a decrease in attraction forces as they are forced apart by water.

3.2 Clay Flowability Test

The successful printing part has the connection between the flowability of the material to extrude flawlessly. The mixture cannot be too soft or too hard. Hence, we need to determine the right mix of the clay. This topic will focus on the material flowability in the extrusion process using 3D print. The measurement of clay mixture flowability includes clay mixture composition, clay density and printing speed.

3.2.2 Clay Mixture Proportion

The water's composition heavily influences the clay mixture's flowability. 1000 g of pug clay is mixed with water used for ceramic clay 3D printing material. Each clay is mixed with a variable amount of water to create the required consistency. The necessary clay is measured in a mass unit (gram) while the water is added in a volume unit (ml). The next step is to combine the two ingredients and stir until the mixture takes on the consistency of clay paste. Each sample will be mixed using an ORIMAS Mixing Machine to ensure the production of the clay paste well-even distributed

Table 1 Mixture of clay-water ratio

Mixture	Clay		Water		Ratio
	(g)	(%)	(g)	(%)	
A	1000.00	70	428.60	30	70:30
B	1000.00	68	470.60	32	68:32
C	1000.00	66	515.20	34	66:34

J. Keeps, 2020 said the best clay to water ratio for ceramic 3D printing is 70:30. However, the limitation of the study was J. Keeps used Kaolin clay as the main material for ceramic 3D printing. To validate the study whether Sayong clay is suitable to use the same ratio as J. Keeps's, the mixture will test with three different kinds of mixture, which is Mixture A, Mixture B and Mixture C. These mixtures vary by its clay to water ratio which are 70:30, 68:32 and 66:34 respectively. The process begins with by weighing the clay for 1kg and slowly pour the water to a total of 426.7g of water. Figure 1 below shows the process of Mixture A preparation. The process repeats with 68:32 and 66:34 clay to water ratio. From the testing, each mixture sample will be printed to determine the best mixture to use as the main mixture for this project.

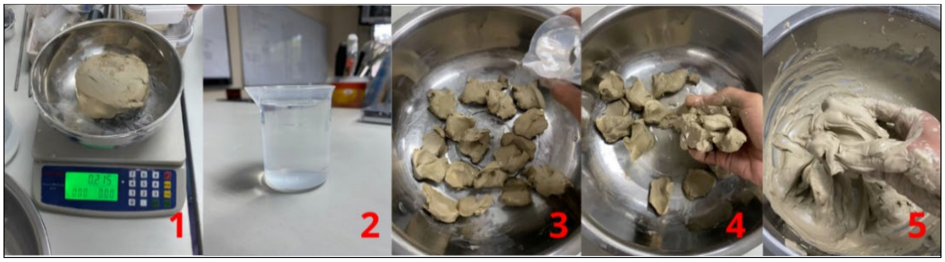

Figure 1 Preparation of clay mixture

3.2.2 Clay Density Test

ASTM D1475 density estimate method provides vital material attributes data. Manufacturing companies use this testing method to evaluate batch-to-batch consistency and product consistency. Density measurements reveal product consistency, solids content, and material composition, making them essential for quality control and formulation. For precision, the test requires a density cup (pycnometer), analytical balance, and room temperature. The 100-mL density cup must be carefully cleaned, dried, and weighed before the test to ensure accurate measurement. Using the cup without clay as the beginning mass, M_1, the sample's net mass is calculated. Next, the clay is carefully added to the density cup to avoid air bubbles, which can significantly impact density measurement. A lid is placed on the cup after filling to prevent spillage or evaporation from changing the sample's volume. Next, an analytical scale with at least 0.001 gramme precision weighs the filled density cup. Mass, M_2 represents the density cup and sample's entire mass while V represents the volume of the Density Cup. Material density, ρ, is computed using the formula:

$$\boldsymbol{\rho = \frac{M_1 - M_2}{V}} \tag{3.1}$$

3.3 Clay Buildability Testing

This study investigated the buildability of a clay mixture by measuring how well it could sustain the weight of its layers as well as those on top. The evaluation of the buildability of the clay mixture by altering slicer settings such as nozzle diameter and thickness of slices. Both tests were conducted utilising the Ultimaker Cura software and Tronxy Moore 1 3D Printer

3.7.1 Nozzle Diameter

By measuring the diameter of the nozzle, the printed component can be obtained. The size of the print also affects the diameter of the nozzle. When the diameter of the nozzle is reduced, the printed object can achieve a higher level of detail. For the nozzle variation test, the standard profile configuration was employed. In this experiment, various nozzles diameter was used which consist of 1.25 mm, 1.75 mm and 2.15 mm with a constant speed of 30 mm/s, and clay mixture B were employed to collect data on (Figure 2).

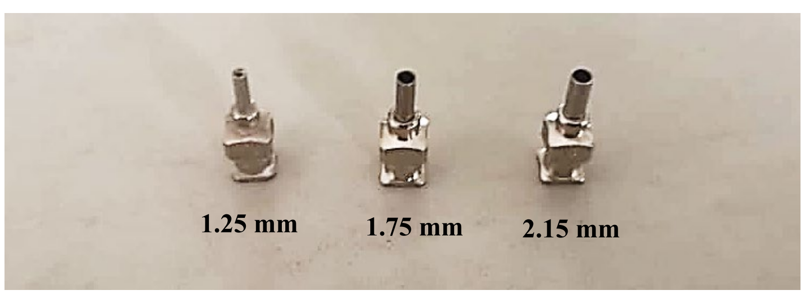

Figure 2: Variation of nozzle size

3.7.2 Layer Height

This study intends to evaluate whether the slice layer height has a substantial impact on the buildability of ceramic clay 3D printing by determining the optimal layer height in proportion to the nozzle diameter for a successful print.

The prepared sample of Sayong clay mixture B was evaluated with various layer height settings of 0.5 mm, 1.0 mm and 1.5 mm with 3 different nozzle sizes and the speed remained constant at 30 mm/s.

4. Results and Discussion

Extrudability and proportional printing tests were performed to determine the optimal consistency for ceramic 3D printing. Using a density test, the density of each clay mixture proportion was calculated. A printing speed test was also conducted to determine the ideal print quality for ceramic 3D printing using tests of nozzle diameter and layer height, the constructability of ceramic 3D printing was analysed. The nozzle diameter test was conducted to evaluate the extruded expansion diameter after printing. In contrast, the layer height test was conducted to evaluate layering quality based on the ratio of layer height to nozzle diameter.

4.1.1 Clay Mixture Proportion Analysis

The amount of mass employed in the test and the variable constant were the same. The clay combination's consistency was assessed by feeling and looking at the mixture due to the challenge of detecting consistency and the lack of a very reliable testing procedure. Sayong clay was combined with various amounts of water. On a Tronxy Moore 1 3D printer, the printing test was carried out using constant parameters of 2.15 mm nozzle size, 30 mm/s printing speed, and 1.5 mm layer height.

Figure 3: Result of the mixture proportion; from left Mixture A, B and C

Figure 3 displays the findings of the comparison of printing experiments for the various clay proportions. All three mixes, A, B, and C, were successfully printed, as shown in Figure 4. Although they have a similar look, they generally differ significantly from one another. The printing worked well for combination C, which has the most water in it, but the first through third layers appear to have been printed unevenly and the layers have been jumbled to the point that the layering effect is no longer discernible. The problem with combination B, which is an uneven layering influence on the first to third layer of the product, is nearly comparable to that with mixture C. Because the content was overly When mixture B was extruded, some of the elements that belonged in the outer layer could be seen in the centre portion of the result. Combination A, which had the least quantity of water in it, produced the best-printed object. There are no noticeable defects to be found in the printing on any of the product's subsequent layers, from the bottom to the top. A higher water content in the mixture will result in faster printing since less material will need to be pushed toward the extruder and less mechanical ram delivery speed will be needed. The amount of water in the ceramic sample has a significant impact on print quality. The surface texture becomes softer as additional water is added to the mixture. The tougher clay will also provide a more broken or shattered surface texture.

4.1.2 Clay Density Test Analysis

The density tests for each clay mixture were performed to determine the specific density of the respective mixtures. These tests are crucial in understanding the material's physical properties, which directly impact its behaviour during subsequent processes such as forming, drying, and firing. The experiment utilized a cup with a specific gravity of 100 ml as the primary measuring tool. The procedure involved carefully filling the cup with the clay mixture to ensure consistency and accuracy. Following this, the filled cup was weighed, and the density was calculated by dividing the measured mass by the fixed volume of the cup. The results of these density measurements provide valuable insights into the composition and homogeneity of the clay mixtures. The data obtained from the experiment are systematically presented in Table 2 below for further analysis and interpretation.

Table 2 shows the clay density measurement result

Mixture	Water Content (ml)	Cup mass (g)	Cup Filled with Clay (g)	Clay mass (g)	Density (ρ)
A	200	198	356	158	1.58
B	200	198	346	148	1.48
C	200	198	336	138	1.38

4.1.3 Model Sampling Test Analysis

Trials and errors process were conducted to make sure the previous setting configuration was suitable enough to be printed for further experiment throughout this project. This experiment was to observe whether the sample will be printed out with 0.2 mm/s, 0.5 mm/s and 1.0 mm/s of extrusion speed at a constant print speed of 30 mm/s to produce high quality products without any defects. The figures below show the outcomes of the sample throughout this experiment.

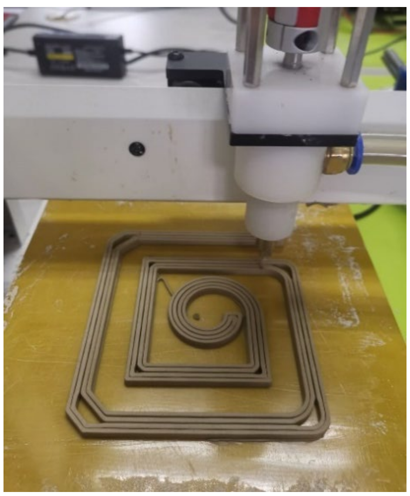

Figure 4(a). Under-extrusion sample

Figure 4(b). Over-extrusion sample

Figure 4(c). Well-extrusion sample

Figure 4(a) above shows that the sample was printed with a condition of under-extrusion. For this sample, the extrusion speed was set to 0.2 mm/s. Although it looks neat and smooth sample but in terms of quality and strength it is quite low because the gaps between those line shows that the sample is not well-printed. This is because the extrusion speed is not compatible with the printing speed that can result as figure above.

In Figure 4(b), it shows that the sample was printed with a few defects. The sample was printed at 1.0 mm/s extrusion speed setting, it shows that there were few clumps during the printing process. This is because the sample was printed under the condition of over-extrusion, which the extrusion speed was a bit too high that it is not balanced with the printing speed which result to several clumps that cannot be accepted as sampling test.

For Figure 4(c) sample, it is a well-extrusion sample as it did not have any defects like gaps, holes or clumps. At extrusion 0.5 mm/s, it can be said that the setting is well-balanced between the extrusion speed and print speed that produce high quality and strong product which can be used for further experiments.

4.2 Clay Buildability Analysis

Clay buildability analysis is a critical aspect of evaluating the performance of clay mixtures in additive manufacturing, particularly in relation to nozzle diameter and layer expansion. Nozzle diameter plays a significant role in determining the resolution and structural integrity of printed layers. A smaller nozzle diameter can enhance detail but may increase extrusion pressure, potentially affecting the uniformity of material deposition. Conversely, larger nozzle diameters improve flow rates and reduce clogging risks but may compromise precision.

Layer expansion refers to the lateral spreading of deposited material after extrusion, influenced by the rheological properties of the clay and extrusion parameters. Excessive expansion can result in dimensional inaccuracies and weakened layer bonding, while insufficient expansion may cause gaps between layers, reducing structural stability. The interaction between nozzle diameter and layer expansion must be optimized to achieve high buildability. This requires balancing material properties, such as viscosity and plasticity, with extrusion conditions, including speed and pressure. A systematic analysis of these factors ensures that the clay maintains its shape during deposition and supports subsequent layers without collapse or distortion, enabling the production of robust and geometrically accurate structures.

4.2.1 Nozzle Diameter Test

The nozzle diameter test was carried out with the aid of LDM 3D printer, namely Tronxy Moore 1 equipped with a mechanical ram delivery. The clay consistency of mixture B was used for the testing, and the print and extrusion speed were held constant at 30 mm/s and 0.5mm/s respectively. Three nozzle diameters were tested to evaluate the width expansion and layer height of the sample extruded after the printing at 5 certain points(Figure 5).

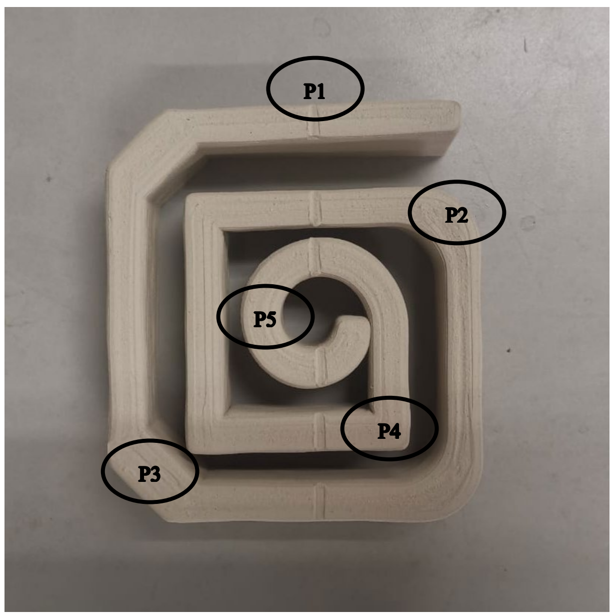

Figure 5. Reference points for width expansion evaluation

Point	Remarks
1	Straight Line
2	Fillet
3	Chamfer
4	Sharp Corner
5	Spiral

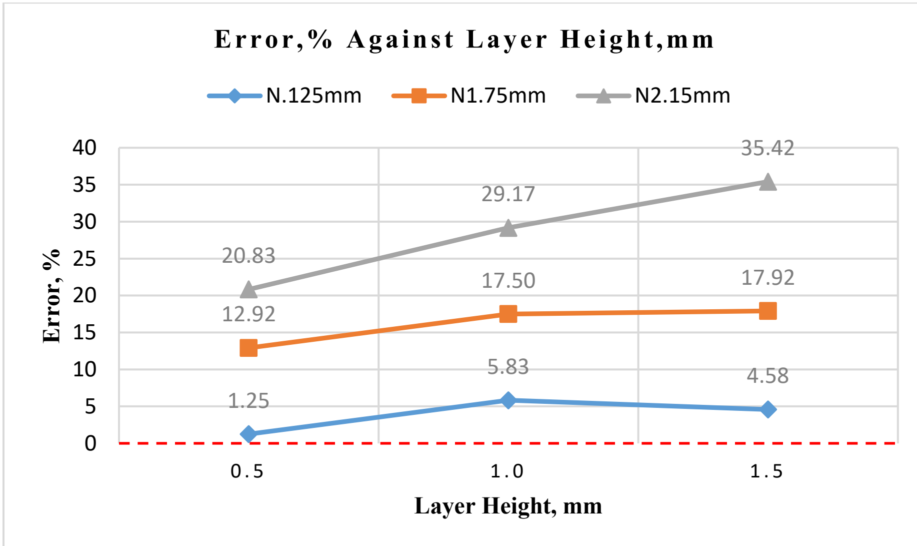

Figure 6. Error against Layer Height Graph at Point 1

Figure 6. shows the error between the 3 sizes of nozzles at certain layer height. The shown value is the average percentage error of the 9 samples. It shows that at the smallest nozzle size at 1.25mm has the least error width offset at straight line which a range between 1.25% to 5.83% which equivalent to 0.1mm to 0.5mm expansion from the actual model at 8mm. For both 1.75mm and 2.15mm nozzles shows a high in value for error. This is because a larger size nozzle means a larger amount of clay extrusion along the printing process that will affect the overall width of the sample.

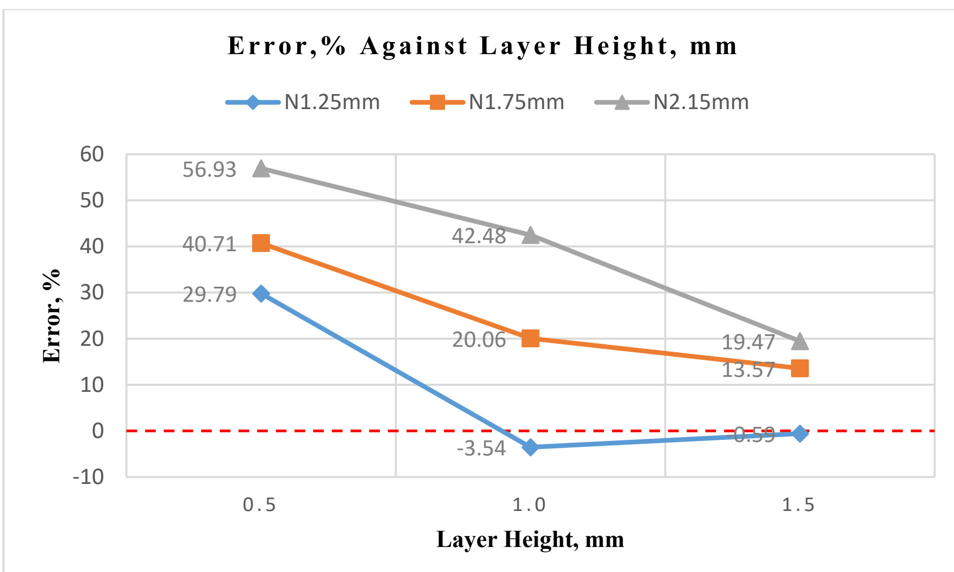

Figure 7. Error against Layer Height Graph at Point 2

Figure 7 above shows the error reading of the sample at Point 2. For nozzle 1.25mm, it has the least error reading ranged between -0.59% to 29.79% which is -0.7mm to 3.37mm offset error from the actual model at 11.30mm. The negative (-) sign indicates the reading is below 11.30mm, however it is still acceptable as successful samples because it has low value of error that can be tolerated. For nozzle 1.75mm and 2.15mm, the error reading shows that the sample printed at Point 2 is up to 50% larger, which is quite high from the actual measurement.

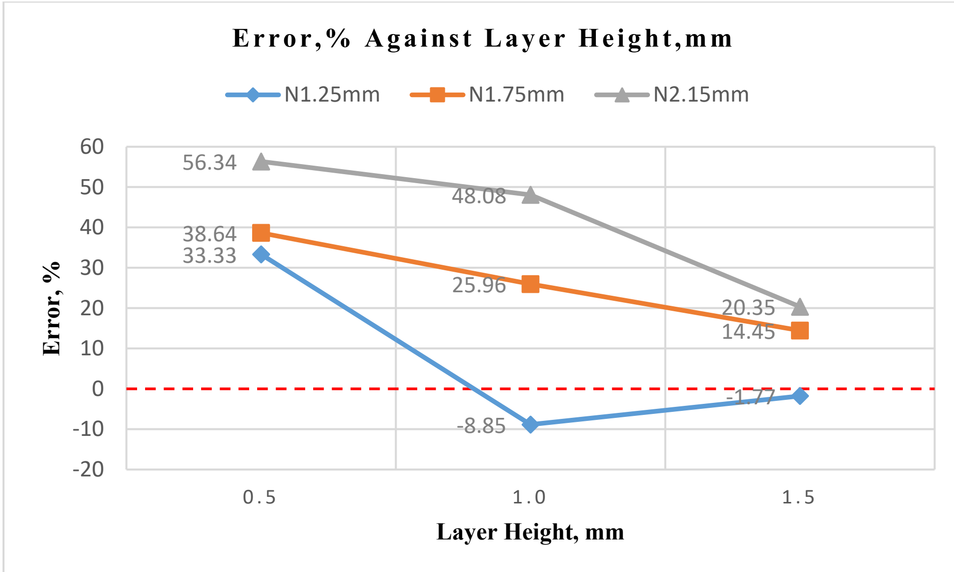

Figure 8. Error against Layer Height Graph at Point 3

The error reading graph of the sample may be seen in Figure 8, which can be seen above. Also displays the least amount of inaccuracy for nozzle 1.25mm, which ranges from -8.85 percent to 33.3 percent, which translates to a -1.0 mm to 3.77 mm offset error from the real model, which is 11.30 mm. Also, for this case, the minus sign (-) indicates that the reading is less than 11.30mm; yet it is still admissible as successful samples since it has a low amount of error that may be allowed. This is because the mistake is quite small. The erroneous reading indicates that the sample produced at Point 3 is almost up to 60% bigger than the actual measurement, which is quite a significant difference when compared to the nozzle sizes of 1.75mm and 2.15mm

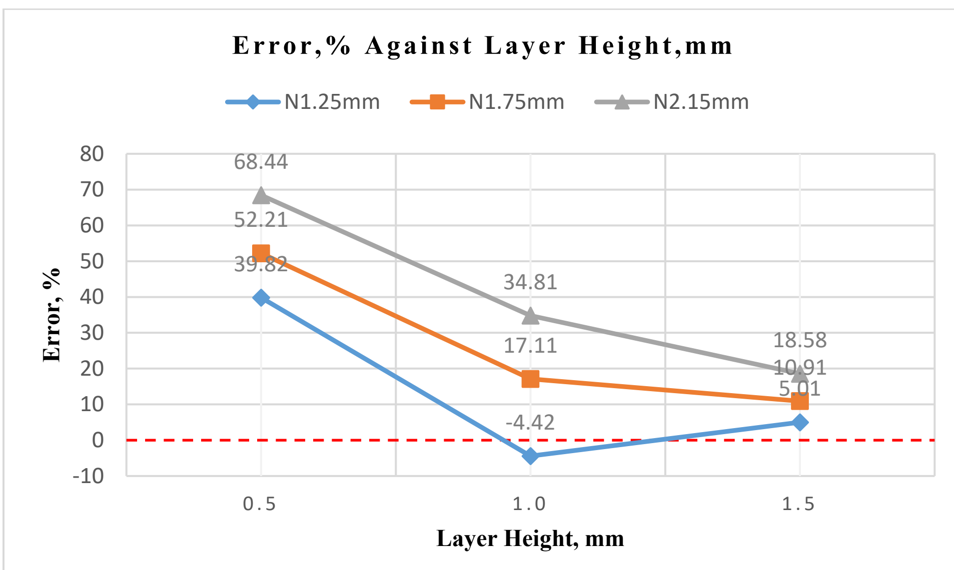

Figure 9. Error against Layer Height Graph at Point 4

Figure 9. illustrates the samples error reading graph. Likewise, nozzle 1.25mm still shows the lowest amount of error, ranging between -4.42% to 39.82%, or an offset error of -0.57mm to 4.50mm from the actual model at 11.30 mm. Though these samples generally meet that standard as valid due to the small margin of error. This is because the error is rather minimal. When compared to the 1.75mm and 2.15mm nozzle diameters, the erroneous reading indicates that

the sample collected at Point 4 is approximately up to 68% greater than the true measurement which is equivalent to 7.73mm.

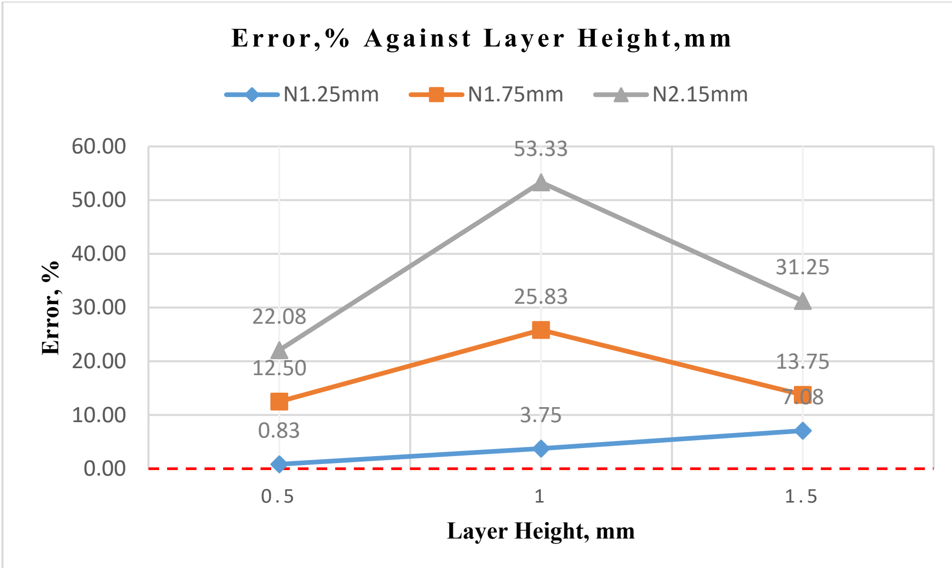

Figure 10. Error against Layer Height Graph at Point 5

Figure 10. illustrates the deviation between the three sizes of nozzles for a given layer height. The shown figure is the mean percentage error across nine samples. It demonstrates that the smallest nozzle size, 1.25mm, has the lowest error width offset at Point 5, ranging from 0.83% to 7.08%, which is comparable to 0.07 millimetres to 0.57 millimetres of expansion from the real model of 8 millimetres. For both 1.75mm and 2.15mm nozzles, the error value is substantial. This is since a bigger nozzle size results in a greater volume of clay extrusion during the printing process, which impacts the total width of the sample.

5. Conclusion and Future Research

This study aimed to evaluate and analyse the physical and material requirements for clay 3D printing, achieving its objectives as outlined in Chapter 1. The first goal focused on assessing flowability by analysing density, printing speed, and clay mixtures, along with the buildability of continuous extrusion of ceramic clay. The second goal involved examining the impact of process parameters on layering quality and faults in printed products by evaluating nozzle diameter and layer height. Finally, the third goal was achieved by printing simple forms to test specific geometric characteristics. The study used Sayong clay, a locally available material in Malaysia, due to its accessibility and cost-effectiveness. Mixture A, with the least water content, yielded the best results, as higher water concentrations reduced clay density. The optimal printing speed was determined to be 30 mm/s for steady extrusion and intact samples. Nozzle diameter tests revealed smaller diameters reduced wall expansion, and a ratio of 1:3 for layer height to nozzle diameter provided smooth, defect-free prints. The findings demonstrate that the material and physical properties examined using the Tronxy Moore 1 clay 3D printer are suitable for clay-based additive manufacturing. Sayong clay shows significant potential for industrial use. Future research should investigate Sayong clay's vitrification, porosity, and hardness at varying temperatures, develop new clay formulas for broader regional applications, and establish advanced sintering and glazing parameters. These efforts will further enhance the capabilities and industrial viability of clay 3D printing.

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