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Differentiated Slip Casting: Producing Variable Thickness Ceramic Tiles with Functionally Graded Plaster Moulds

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Abstract: This paper introduces a method that enhances the traditional slip casting technique's potential to fabricate ceramic objects with variable thickness. The variability depends on the different filtration rates offered by plaster moulds of varying densities. Two sets of experiments are presented. They focused on identifying (1) the maximum workable density range of moulds made from plaster of Paris and (2) the range of thickness in the resulting ceramic casts. This was accomplished by creating four square flat moulds with different gypsum/water (G:W) ratios and their corresponding casts. Based on these findings, the second set of experiments focused on assembling graded plaster moulds with variable densities (G:W 1:3 to 2:1), resulting in ceramic tiles exhibiting a thickness gradient of 2 mm. These results suggest the possibility of producing double-curved ceramic objects (e.g., custom ceramic tiles or sanitaryware) with graded thickness, tailored to their desired structural and functional performance.

Keywords: slip casting; functionally graded plaster; variable thickness; ceramic tiles; variable densities; plaster of Paris

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1. Introduction

The process of slip casting clay in plaster moulds is one of the oldest material forming techniques, and it remains widely popular both in artistic and industrial ceramic production worldwide. The slip casting technique relies on the interaction of two material composites: (a) clay/water (clay slip), and (b) gypsum/water (plaster). The interface between these two composites determines the performance (e.g., thermal, mechanical) of the resulting ceramic parts. Specifically, the porosity of the plaster mould dictates the filtration rate of the clay slip, and ultimately, the wall thickness of the resulting clay body. Traditionally, ceramic bodies produced by slip casting exhibit constant wall thickness [1,2]. Under uniform material and environmental conditions, wall thickness has traditionally been found to be directly proportional to the square root of the casting time [3], but in some cases, it has also been found to follow a linear relationship [4].

Since the 1990s, the emergence of functionally graded materials (FGMs) has provided the opportunity to design and fabricate novel materials with a non-uniform microstructure across their volume. Among the several methods behind the production of FGMs, some rely on the slip casting process. Centrifugal slip casting and sequential slip casting have been extensively investigated for the production of spatially graded ceramic–metal or ceramic–ceramic composites [5]. Both methods depend on the variability of slip composition, while they both rely on plaster moulds of uniform density [6].

To the authors' current knowledge, no previous work has addressed the possibility of fabricating functionally graded plaster moulds for the production of single-material ceramic objects with variable thickness.

Building on the fundamental principles that regulate the slip casting process, we propose a novel methodology to fabricate ceramic bodies with a controllable wall section thickness by solely controlling the material composition of the plaster moulds. Specifically, we explore the correlation between the density of the plaster mould and the rate of absorption of the ceramic slip [7,8] by exclusively adjusting the moisture content of the plaster mix [9], while keeping the settling time and slip composition parameters constant.

By experimenting beyond the density range of plaster mixtures recommended by the provider, we aim to maximise the impact of the plaster mould's density on the thickness of the resulting clay cast. Finally, we discuss possible applications of this differentiated slip casting technique, introducing a new fabrication method for creating ceramic tiles with controlled variable thickness.

2. Materials and Methods

The first set of experiments of this research are based on the traditional slip-casting technique, using plaster moulds and clay slip. The focus lies on investigating the boundaries of workable densities, low and high, for the production of plaster moulds, and their impact on the thickness of the resulting slip-casted clay bodies.

This is demonstrated through the production of a series of planar plaster moulds with uniform size (120 × 120 × 50 mm) but variable density. Subsequently, we document the production of planar clay samples by slip casting on the mentioned plaster moulds. By analysing the results of this set of experiments, we identify the widest usable plaster density and cake thickness range.

In all experiments, the room conditions were kept constant. Specifically, the relative humidity was kept at 39% and room temperature was set to 24 °C. In addition, the type of clay slip and the type of plaster, as well as the casting time, remained constant.

2.1. Plaster Moulds

The plaster moulds consisted of two ingredients: (a) gypsum powder and (b) tap water at room temperature (24 °C). The gypsum powder used was a commercial product (Modelling gypsum produced by Krone, Germany) of calcium sulphate hemihydrate ($CaSO_4 \cdot 0.5H_2O$, "Plaster of Paris"). According to the producer's datasheet, the above powder consisted of finely ground gypsum binder from selected natural white gypsum stone without factory additives. The set time of the above product was 15 min for the recommended gypsum powder/water (G:W) weight ratio, spanning from 5:3 to 2:1. Aiming to identify the boundaries of possible densities, we expanded the range of these mixing ratios to the maximum workability, successfully experimenting in the domain between 1:3 and 3:1. Within this range, we selected four different G:W ratios to test (1:3, 1:1.3, 2:1, and 3:1). The first two ratios were well below the provider's suggested range (i.e., with an excess of water), the third was the maximum ratio of the suggested range, and the fourth was above the suggested range (i.e., an excess of gypsum powder).

To achieve these ratios, the quantities of both ingredients were measured with an electronic scale and hand-stirred with a spatula to form the different plasters. Based on prior research, it is known that the higher the moisture content of the plaster mix, the lower the mechanical strength of the mould [10]. As expected, the tested range of mixtures exhibited a wide viscosity range: low viscosity for smaller G:W ratios and high viscosity for larger ratios. Depending on the viscosity of each mixture, both the required mixing and working time were found to differ significantly, spanning from 1 to 10 min. The mixtures were then either poured or plastered into master moulds made of water-resistant laminated wood. After 24 h, all of the plaster mixtures were hardened enough to be de-moulded, and subsequently were left to air-dry at the above-specified room conditions. Based on empirical evidence, all of the mixtures were dry within a period of 12 days.

2.2. Clay Casts

After the four above-mentioned plaster mould variants were air-dried, they were used for the (simultaneously conducted) slip casting of four clay samples. According to p. 187 from [11], to ensure that no moisture was trapped in the moulds, they were additionally dried in a kiln at 60 °C. Based on empirical testing, 3 h of kiln drying was needed to reach a full dehumidification of the moulds. All plaster moulds used in the presented experiments were used at early stages in their service time (fewer than 80 casts) [12].

The clay slip used in all of the presented experiments was a commercial product (manufactured by Keramikbedarf Ing. Skokan GmbH, Vienna, Austria). According to the producer's datasheet, the chemical composition of the dry clay powder of the casting mass in oxide equivalents is 75.0% SiO_2, 19.8% Al_2O_3, 1.4% TiO_2, 0.9% Fe_2O_3, 0.2% CaO, 0.3% MgO, 2.2% K_2O, and 0.1% Na_2O (clay powder Nr. 208 provided by Goerg & Schneider, Boden, Germany). Besides the clay powder, the slip contains 37 wt.% water and two deflocculants/liquefiers: 0.1 wt.% of Dolaflux B and 0.1 wt.% of Giessfix 162 (both produced by Zschimmer & Schwarz, Lahnstein, Germany). Before casting, the slip was stirred with an electric rotating mixer for 10 min. Then, it was carefully poured to fill the volume of the laminated wooden formworks that contained the planar plaster moulds. The settling time of the slip was kept constant for all of the experiments, at 60 min. This duration was selected based on prior traditional slip casting experiments in order to achieve a cast thickness similar to the one of standard clay tiles (approximately 6 mm).

After 60 min, the excess clay slip was manually poured off the formwork and flipped upside down for 5 min to drip the slip residues. The mould then was placed upright. After approximately 15 min, the wooden walls of the formwork were carefully removed to reduce adhesion-related tensions between the clay and the wooden walls. While in the leather-hard state, the clay body was removed from the plaster mould and placed on a flat plaster surface to dry, at room conditions, until mass constancy was reached. Our empirical evidence shows that, given the size of the clay samples at that moment and the mentioned room conditions, 72 h was typically required for a complete drying. As soon as the clay bodies had dried, they were fired at 940 °C in a numerically controlled kiln (Nabertherm, model P470) with the following sintering temperature cycle conditions: from 25 °C to 600 °C at a 150 °C/h rate increment; from 600 °C to 940 °C at a 300 °C/h rate increment; once 940 °C was reached, the program would stop and wait to reach room temperature at a natural decrement rate. The temperature cycle was selected based on an established laboratory routine, with heating rates in the range recommended for comparable materials in "*Ceramic technology and processing: a practical working guide*" [11] by King, pages 280–281.

2.3. Evaluation Methods of the Differentiated Slip Casting and Material Characterization

To evaluate the correlation between the plaster mould density and obtained wall thickness of the clay samples, the following three methods were used.

The first method returned the mass data of both plaster moulds and fired clay bodies by electronic scale measurements (0.1 g accuracy).

The second method investigated the pores and density characteristics of both plaster moulds and fired clay casts. For that, small fragments (~10 × 10 mm^2) were extracted from the centre of the samples. In particular, pore characterization of the cured and dried plaster mould materials and fired clay specimens were determined via mercury intrusion porosimetry (Thermo Scientific Pascal 140/440), using intrusion pressures up to 400 MPa. Before analyses, the specimens were dried at a temperature of 50 °C. Additionally, bulk density, skeletal density, and apparent porosity of fired clay specimens were determined using the water immersion method, following EN 623-2 [13], using a minimum number of three specimens per sample.

The third method returned a high accuracy measurement of the fired clay samples' thickness via 3D scanning. The presence of distributed material imperfections on the top surface of the clay samples led to the discarding of simpler measuring tools (e.g., callipers, gauges). In the attempt to render a more comprehensive description of thickness distribution and variability across the whole sampled area, a 7 degrees of freedom hand-held Metris MCA Measuring Arm with 0.05 mm probe tolerance was used. The measurement process started by safely positioning each specimen to allow the simultaneous acquisition of their top and bottom surfaces. Each measurement resulted in a dense 3D point cloud. Each point cloud was clipped to a uniform size (75 × 75 mm^2) and not further post-processed. Each clipped point cloud was organised as a pair of top and bottom surfaces, approximately 20,000 points large. For each specimen, a single thickness value was computed as the mean value of a closest-point distance calculation from each of the 10,000 single points of the top surface towards the bottom surface. A standard deviation value was associated to each mean thickness value. In-house developed software was utilised for point-cloud processing and thickness measurement.

3. Results

Utilizing the four above-mentioned square planar plaster moulds with different densities, named according to their G:W ratios (1:3, 1:1.3, 2:1, and 3:1), the structural characteristics, mass, and thickness data of all relevant parts (plaster moulds and/or fired clay casts) were evaluated.

3.1. Structural Characteristics

3.1.1. Plaster Moulds

In the first stage of the structural characterization, porosity characteristics of the four plaster mould materials were evaluated using mercury intrusion porosimetry of their fragments (around 0.1 to 0.2 g each) (Figure 1). The results show that mould materials prepared from plaster slips containing higher amounts of water (1:3 and 1:1.3) exhibited significantly lower densities and, consequently, higher porosities, with an additional variation in pore opening size (Figure 2).

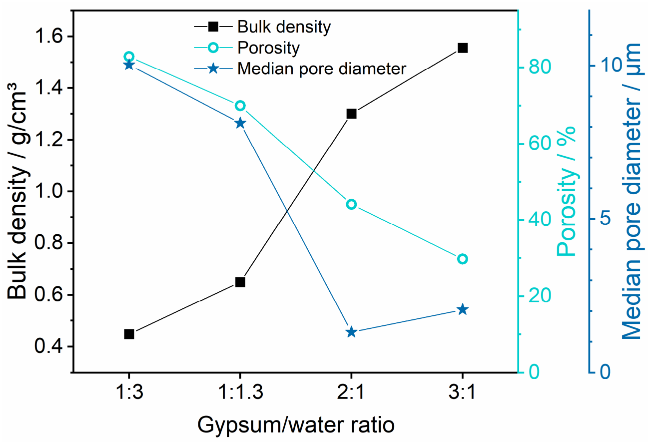

Figure 1. Bulk density, porosity, and median pore opening diameter of plaster mould materials prepared from varying gypsum/water ratios, determined via mercury intrusion porosimetry.

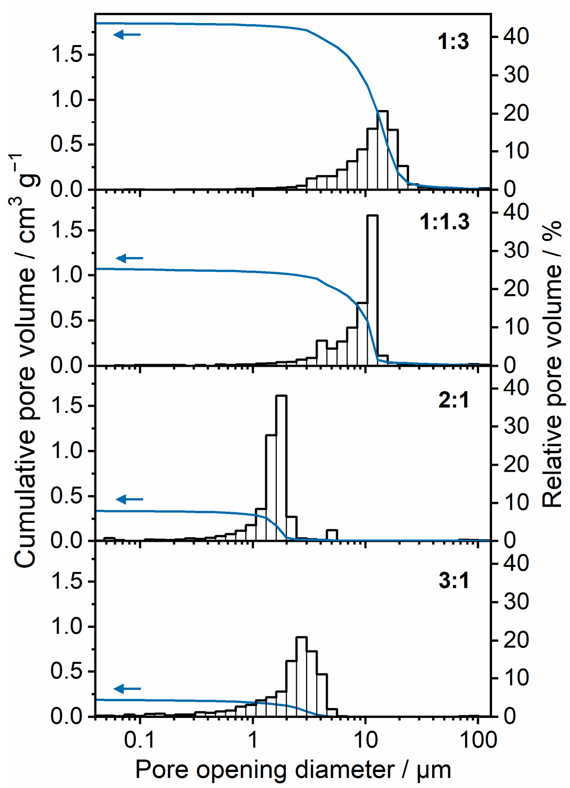

Figure 2. Pore opening diameter distribution of plaster mould materials prepared from varying gypsum/water ratios, determined via mercury intrusion porosimetry.

With an increasing amount of water in the starting slip, both porosity and pore size within the cured and dried plaster moulds increase. This may be explained by network formation during hydration, with excess water being removed during the subsequent drying, thus retaining more and larger pores within the structure. Based on these results, differences in cake build-up during casting may be expected due to pore size-dependent variations in capillary forces, with plaster moulds 2:1 and 3:1 likely to provide an accelerated increase in cake thickness compared to plaster mould materials 1:3 and 1:1.3.

3.1.2. Fired Clay Casts

After firing the four clay samples, density characteristics and total porosity of fragments (weighing between 0.4 and 3.0 g) were determined by the water immersion technique (Table 1). Firing density, apparent density, and porosity appear to be independent from the plaster material composition used during casting. These results are also confirmed by mercury intrusion porosimetry, which also show comparable pore sizes within the four samples (Figure 3). Light optical micrographs of the fracture surfaces of fired specimens (Figure 4) do not indicate any significant differences between samples.

Table 1. Density and total porosity characteristics of fired clay samples cast in plaster mould materials prepared from varying gypsum/water ratios, determined by the water immersion technique.

Sample	Bulk Density (g cm^{-3})	Apparent Density (g cm^{-3})	Apparent Porosity (%)
3:1	1.70 ± 0.01	2.71 ± 0.01	37.3 ± 0.2
2:1	1.70 ± 0.01	2.71 ± 0.01	37.3 ± 0.1
1:1.3	1.70 ± 0.01	2.72 ± 0.01	37.4 ± 0.1
1:3	1.69 ± 0.02	2.71 ± 0.02	37.5 ± 0.5

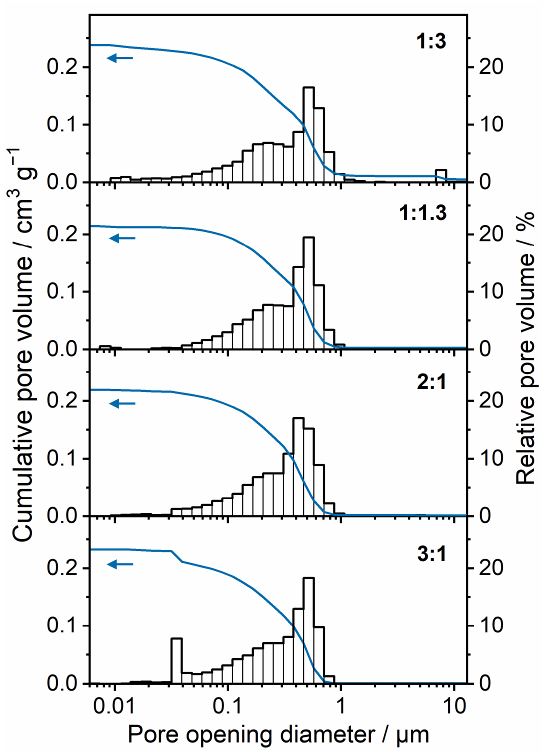

Figure 3. Pore opening diameter distribution of fired clay samples cast in plaster mould materials prepared from varying gypsum/water ratios, determined via mercury intrusion porosimetry.

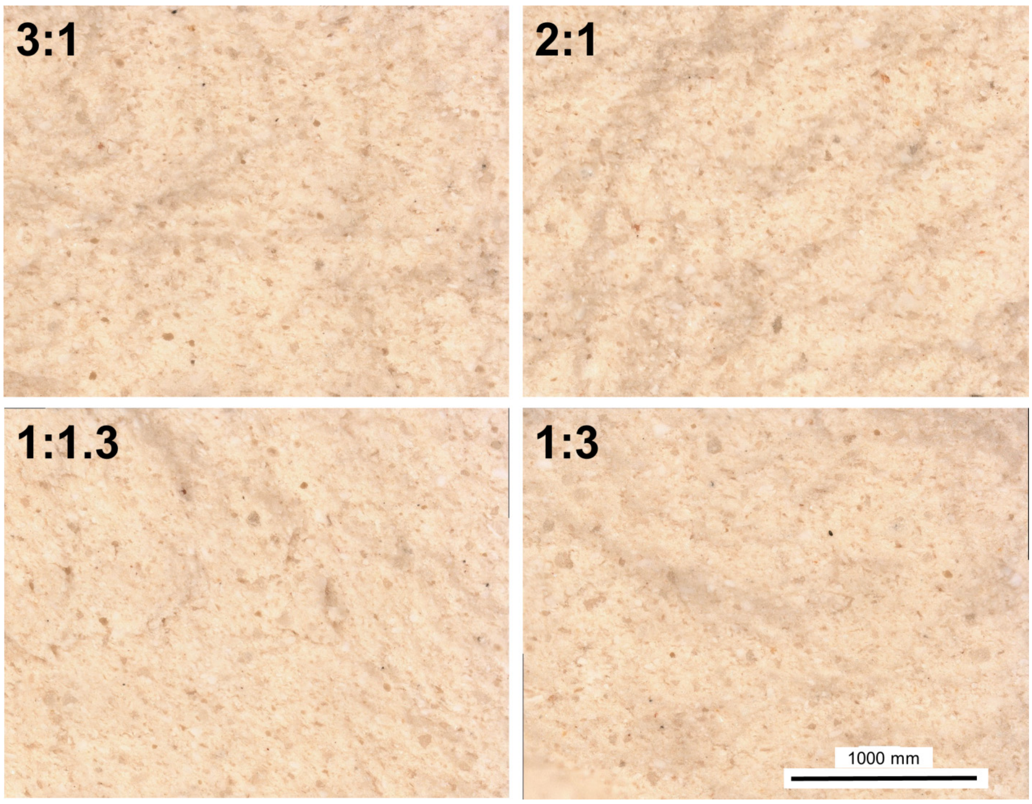

Figure 4. Light optical micrographs of fracture surfaces of fired clay samples cast in plaster mould materials prepared from varying gypsum/water ratios.

Based on these results, the density, porosity, and microstructure of the fired clay bodies appear to be primarily affected by the firing process, while the porosity or density of the plaster mould material does not seem to be a relevant factor. Therefore, these results show that the variation in mould material composition indeed provides a viable means of controlling the cake formation process during casting. Consequently, the morphology of the resulting green body, i.e., differences in the green body microstructure are effectively eliminated during firing, resulting in a highly uniform material structure.

3.2. Water Absorption and Casting Performance

The mass of the four plaster moulds was measured with an electronic scale immediately before and after the clay slip casting process to determine the amount of water absorbed by the mould. The results (Figure 5, blue) indicate that for the first three samples (1:3, 1:1.3, 2:1) the water absorption increases linearly according to the mould density. However, the densest mould, 3:1, showed reduced water absorption in comparison to mould 2:1. A comparable linear increase is observed for the resulting mass of the four clay casts in both the dried green and fired state (Figure 5, purple and red, respectively). Finally, the cast morphology after 60 min simultaneous slip casting using the four mould material compositions is shown in Figure 6, illustrating green bodies, the moulds' water absorption, and slip excess level.

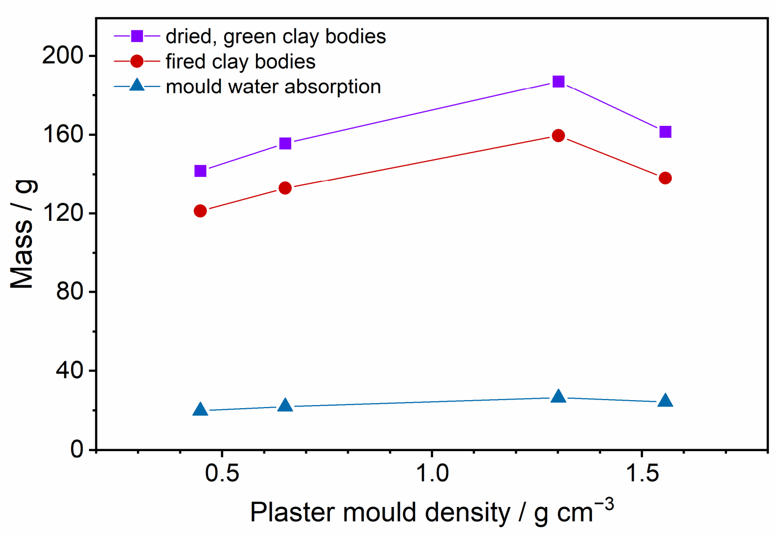

Figure 5. Correlation between plaster mould densities and cast/mould masses. Mass data of the four plaster clay casts of different G:W ratios (1:3, 1:1.3, 2:1, 3:1), in both dried green (purple line) and fired state (red line), as well as water absorption (blue line) of the four plaster moulds after the slip casting. Casting duration 60 min, casting area 120 × 120 mm^2.

3.3. Thickness

The thickness of each fired sample was measured by means of a 3D laser scanning method, thus yielding additional information on thickness variation and distribution. By calculating the arithmetic mean value of all of the point-to-point distances for each sample tile, a global thickness variation among the four samples is clearly identified. A standard deviation value σ was coupled to each mean value to describe the thickness distribution across each specimen surface. Sorting the values according to increasing G:W ratios (1:3, 1:1.3, 2:1 3:1), the resulting thicknesses are: 5.67 ± 0.23 mm, 6.26 ± 0.28 mm, 7.63 ± 0.30 mm, and 6.64 ± 0.27 mm. The mean values are in line with the previously observed trends, confirming that resulting thicknesses of the first three plaster moulds follow a linearly ascending trend, with the clay body obtained from the mould composition 3:1 again exhibiting decreased thickness values. Figure 7 renders the variability in thickness distribution across the surface of the four clay samples and quantifies it within a standard deviation (σ) range between a maximum of ±0.3 mm (mould 2:1) and a minimum of

±0.23 mm (mould 1:3) around the respective mean values. Such σ values confirm and quantify a certain consistency in local unevenness of the top surface of the specimens which emerged during the manual process of slip removal.

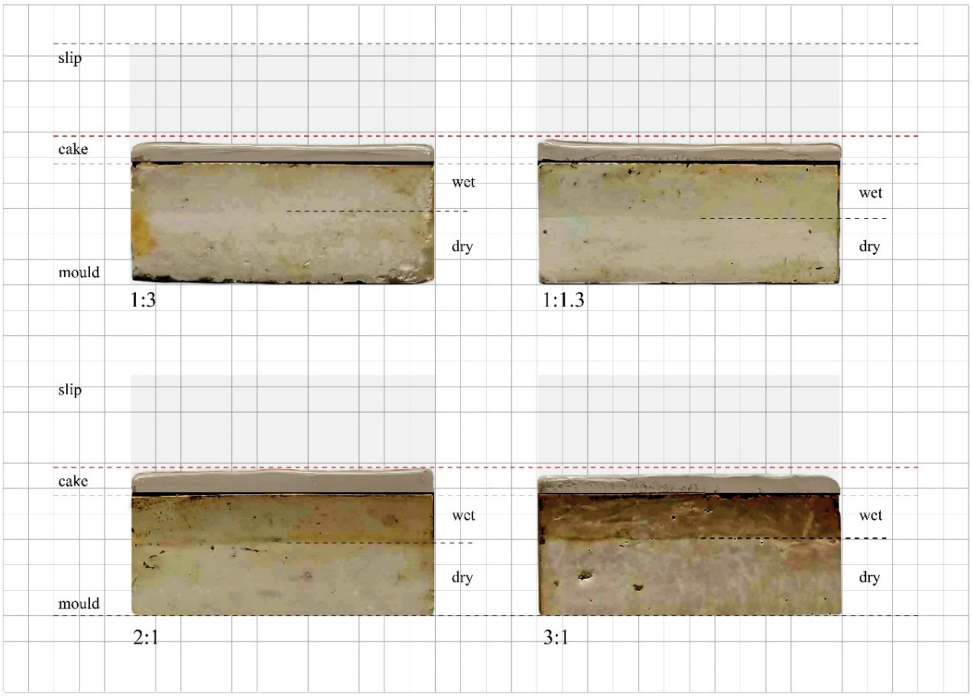

Figure 6. The four plaster moulds with various densities with their corresponding clay cakes in wet green state. The depth of the water absorption of the moulds is indicated with a dashed line and is divided into wet and dry. The slip level during casting is highlighted with grey colour. Casting duration 60 min. Square grid represents 1 cm.

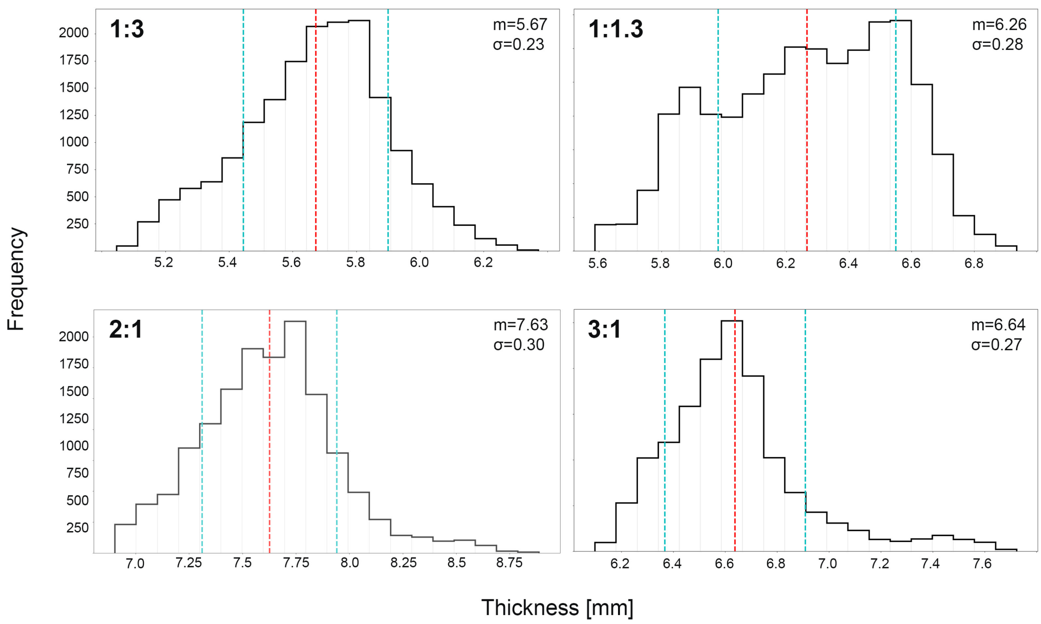

Figure 7. Thickness values distribution in mm of the 4 fired clay samples (75 × 75 mm^2) extracted from 3D point cloud data. The mean thickness values (red) of the samples range between a minimum of 5.67 mm in mould 1:3 and a maximum of 7.63 mm in mould 2:1. Standard deviation values (blue) quantify the variability in thickness distribution across the samples' surface in a range between ±0.30 mm in mould 2:1 and ±0.23 in mould 1:3.

4. Discussion

Completing the evaluation of the structural characteristics of both plaster moulds and clay casts, as well as the thickness investigation via a 3D scanning technique, it can be concluded that within the range of plaster mould G:W ratios between 1:3 and 2:1, a thickness variation of the clay casts of approximately 2 mm can be achieved (Figure 8) within the typical slip casting time of 60 min used in this work.

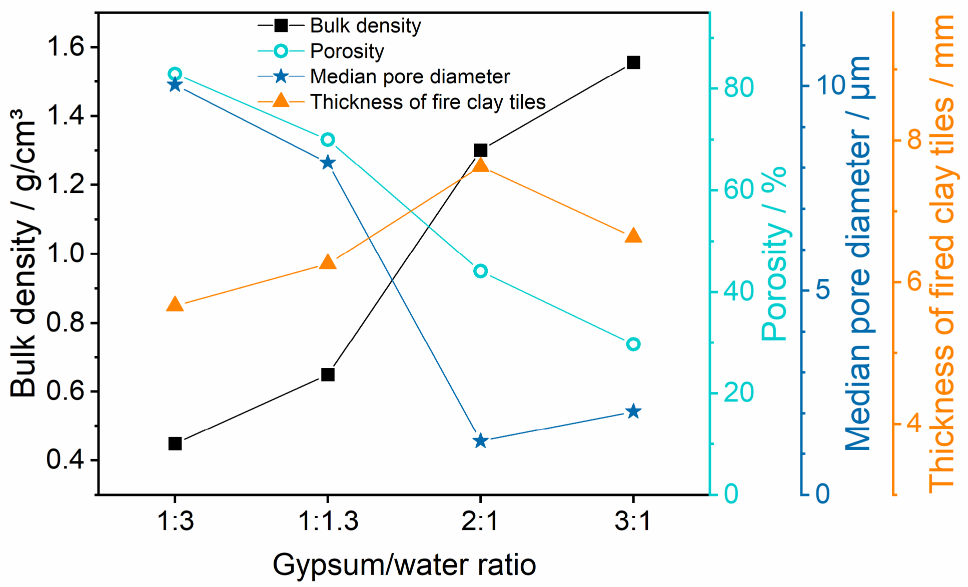

Figure 8. Bulk density, porosity, and median pore opening diameter of gypsum mould materials prepared from varying gypsum/water ratios, determined via mercury intrusion porosimetry, as well as the average thickness values of the respective clay casts after firing.

Based on this range, we produced variable thickness clay tiles by slip casting on composite plaster moulds, assembled from individual moulds with different plaster densities. The range of utilised plaster densities is shown in Figure 9. To demonstrate the possibility of utilising functionally graded plaster moulds with both variable density and spatial configurations, we present two examples of variable thickness clay tiles: one linearly graded tile (measuring 490 × 150 mm^2) and one radially graded tile (150 × 150 mm^2).

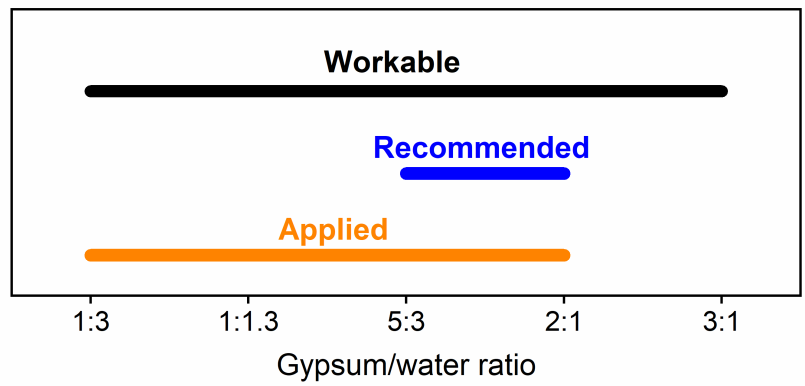

Figure 9. Ranges of gypsum/water ratios considered for plaster mould production. In black, the maximum workable range (higher or lower ratios resulting in excessively thick or thin plaster mixes). In blue, the recommended optimal range, as specified by the gypsum producer. In orange, the applied range ultimately utilised for the production of the thickness gradient (1:3 for the thinnest cake, 2:1 for the thickest).

4.1. Production of Thickness-Graded Tiles

The linearly graded mould was produced by means of one adjustable formwork for plaster casting. The mould design consisted of five equally sized rectangular sections characterised by five consecutive plaster G:W ratios: 1:3, 1:2, 1:1.3, 2:1, and 3:1. It was produced in five subsequent steps, by pouring one plaster mix after the other (starting from the less viscous to the more viscous) in the formwork by enlarging its size at each pouring step (Figure 10, left). The working time of the different mixtures was variable, according to their viscosity, and spanned from 2 min (for the highest viscosity mix) to 15 min.

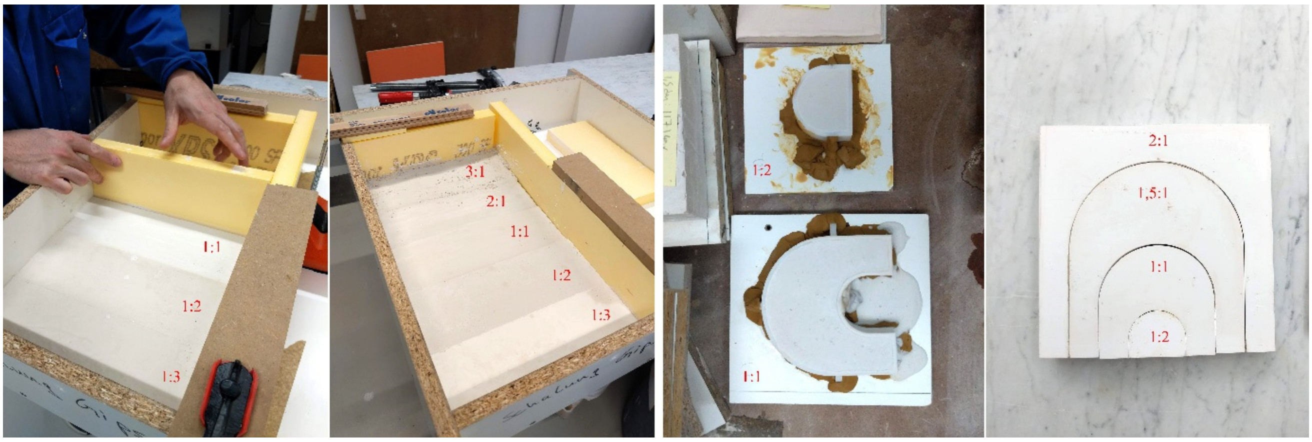

Figure 10. Production process for the two gradient plaster moulds. **Left**: Linear gradient mould produced by subsequent casting of parts with various densities in one master mould. **Right**: Radial gradient mould produced by combining four individual parts with different densities.

The radially graded mould consisted of four sections with different G:W ratios of 2:1, 1.5:1, 1:1, and 1:2. Another fabrication process was used to make this graded mould. First, four 3D printed ABS interlocking master moulds were produced and subsequently cast with the different plaster mixtures. After each mixture was poured and cured in each respective master mould part, it was carefully demoulded. Finally, the four individual plaster parts were assembled to create the combined functionally graded mould (Figure 10, right). The same slip-casting processes as the ones described in Section 2.2 were used for both of the graded tiles. While in the leather-hard state, both clay tiles were trimmed from the edges to facilitate the 3D scanning procedure. Subsequently, they were dried and fired with the same parameters described in Section 2.2.

4.2. Thickness Gradient Evaluation

The top and bottom surfaces of the two fired graded tiles were 3D scanned and used to extract thickness values from 3D point cloud data (as described in Section 2.3). Their original size was reduced due to the requirements for the scanning setup fixtures (scanned linear tile: 375 × 65 mm^2; scanned radial tile: 75 × 150 mm^2). The measured data were colour mapped directly onto the scanned geometry to visually render the thickness variations. The linear tile showed an average thickness range spanning from a minimum of 6.49 mm to a maximum of 7.94 mm, while the average thickness of the sections of the radial tile ranged from 5.15 mm to 6.15 mm (Figure 11). The measured data, rendered as a grayscale heightmaps (Figure 11B,E), clearly confirmed a global thickness variation of both graded tiles in correspondence to the variably dense plaster mould sections. The measured data rendered as a colour-coded contour maps (Figure 11C,F) revealed a smooth clay thickness gradient across the different plaster mould regions.

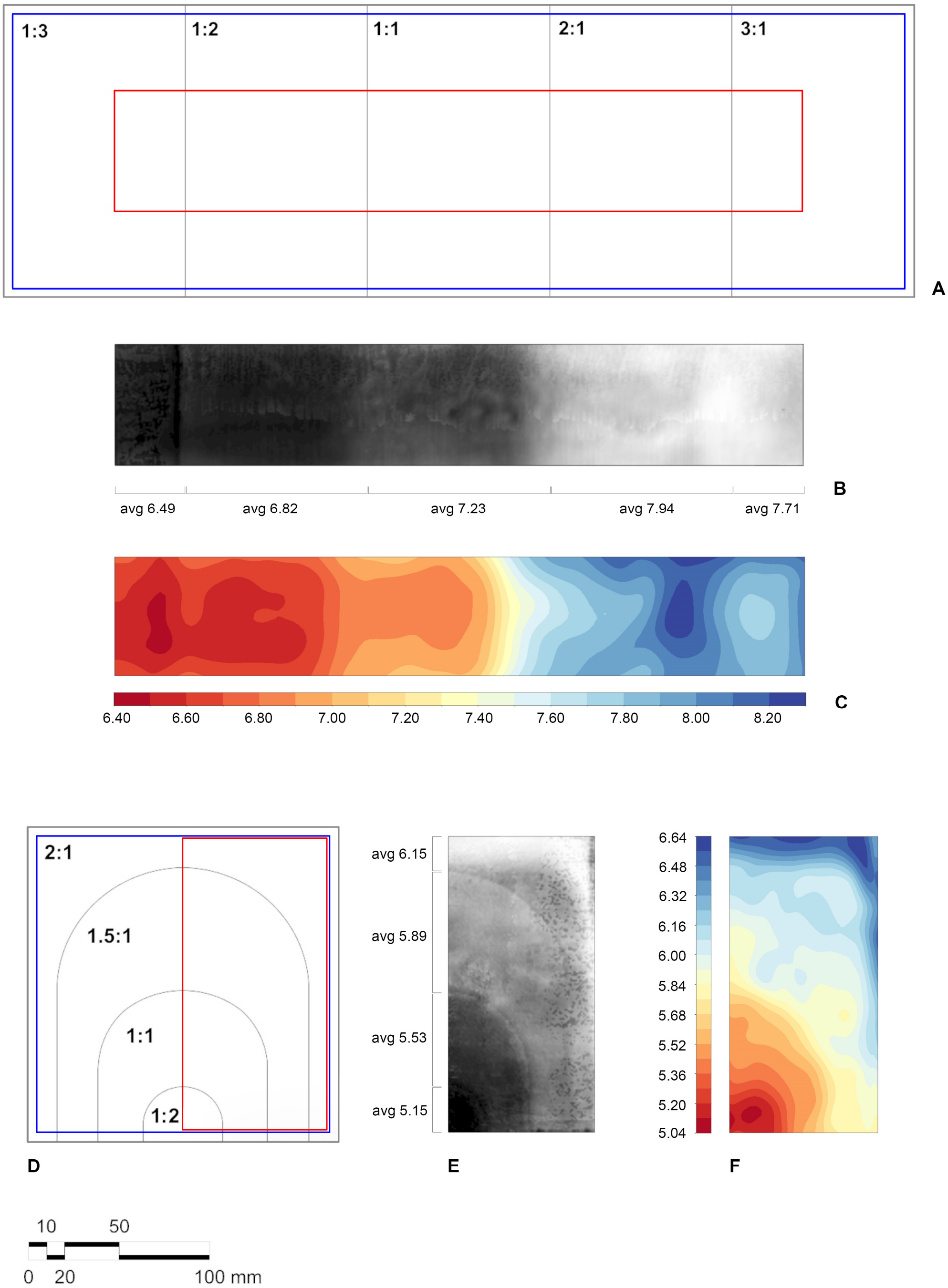

Figure 11. Visualization of the measured thickness values of the graded tiles: linear tile (**A**–**C**) and radial tile (**D**–**F**). For each tile we show size and position (**A**,**D**) of the 3D scanned surface (red) relative to the entire clay tile (blue) and to the plaster mould (black). The measured thickness values are rendered as: (1) grayscale height map with thickness values averaged per mould-area (**B**,**E**) confirming the correlation between plaster mould density and ceramic tile thickness; (2) colour-coded contour map at constant height steps (**C**,**F**) revealing a smooth clay thickness gradient across the variably dense plaster mould regions.

5. Conclusions

Our research introduces a method for fabricating ceramic objects with variable thickness using differentiated slip casting with functionally graded plaster moulds. Through experimentation and analysis, we demonstrated the effectiveness of this method in achieving smooth controlled thickness gradients in ceramic tiles. In particular, we observed that the variability in plaster density does not compromise the density uniformity of the fired clay, indicating the robustness of the technique. The ability to create thickness gradients opens avenues for tailoring ceramic performance, including thermal and mechanical properties, in various applications.

Despite these achievements, we acknowledge several limitations. The variations in ceramic thickness observed in the sample tiles may not perfectly translate to the graded tiles due to challenges in controlling the slip viscosity. Additionally, surface unevenness remains a concern that requires technical refinement. Finally, the high brittleness of the less dense plaster moulds (e.g., 1:3), as well as the complexity of the multiple parts of the mould (e.g., radial), could lead to challenges in the production scalability.

Future research could focus on the fabrication of more volumetric objects to explore further design possibilities. Furthermore, collecting data on plaster and clay slip viscosity would significantly improve the fabrication process. Finally, additive manufacturing technologies would lead to further developments of our novel slip-casting application (e.g., formwork-less and single-step graded gypsum mould production), as well as to novel 3D printing hardware solutions (e.g., close-to-the-nozzle mixing).

Author Contributions: E.B. and M.P.: Conceptualization; Data Curation; Formal Analysis; Investigation; Methodology; Project Administration; Resources; Software; Supervision; Validation; Visualization; Writing—Original Draft; Writing—Review & Editing. F.H.: Investigation. T.K.: Formal analysis; Resources; Visualization; Writing—Review & Editing. M.K.: Writing—Review & Editing. All authors have read and agreed to the published version of the manuscript.

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