**Eskisehir Technical University**
**Materials Science and Engineering Department**

# MLZ 331

# *Materials Processing Laboratory-I*

## 2024 – 2025 FALL

**Course Instructors**

Prof. Dr. Ferhat KARA
Prof. Dr. Semra KURAMA
Prof. Dr. Alpagut KARA
Res. Assist. Dr. Kübra GÜRCAN BAYRAK
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**Course Coordinators**

Res. Assist. Dr. Kübra GÜRCAN BAYRAK

# GENERAL INSTRUCTIONS FOR
# MLZ 331 MATERIALS PROCESSING LABORATORY-I

1. Because the experiments are not synchronized with the classroom lectures (i.e., Ceramic Processing), **it is extremely important that you read each experiment and basic references prior to the lab.** The lab instructor will ask general questions during the lab to test your understanding of the lab.
2. The data, calculations, observations, results and graphs are to be recorded directly, into the **report sheet.**
3. **At the beginning of experiments**, there will be **a quiz composed of 2-3 questions.**
4. **Two reports** will be submitted throughout the term. **The first report** will be **a formal report**. Detailed instructions for preparing the report are provided under the section titled **'15. INSTRUCTIONS OF REPORT I'**. The second report will consist of Questions, with two questions from each experiment. Instructions for this part are also provided under the section titled **'16. INSTRUCTIONS OF REPORT II'**
5. The submission dates for these reports are indicated in the schedule below. These reports will be a digital copy (should be PDF document), and it will be sent to **www.turnitin.com**. Upload your files (firstly, you need to be logged in) with **45528053 class ID and Mlz331 enrolment key**. Files should be uploaded by the name format of **GroupName_Student Number_Name&Surname**
   (For exp.; **GroupA_12345678910_AhmetYılmaz**).
6. Each experiment has **dedicated sections in the report sheets, which will be filled out synchronously during the experiments.** These report sheets are to be submitted to the experiment instructor of that **week before the next week's experiment.** Those who do not submit **a report sheet for the relevant experiment will not be evaluated for the answers to the relevant experiment questions in the Question section to be evaluated within the scope of Report II.**
7. Students are responsible to submit their reports to Turnitin class **on the same day until 18.00 a week after the experiment**. The lab instructor will grade the reports within a week and will post the results. If you wish to discuss the grade, make an appointment to see the lab instructor at her convenience. A copy of the graded reports will be handed to you upon your request if needed.
8. Reports must be handed in on time; **otherwise 10% will be deducted from the mark for each day late.** If there are extenuating circumstances for a report not being completed on

time or for not attending a lab, the student should phone or make an appointment with one of the course instructors.

9. The nature of working in groups implies that there should be cooperation and discussion between members of the group and the lab instructor. It is, however, expected that when students prepare their reports, that they do so individually using their own words and interpretation. Plagiarizing or blatant copying of a report or reference will result in an automatic zero for that lab for the first offense. A second offense will result **in an automatic FF grade for the course.**
10. Students must attend each lab on the specified date unless arranged differently with the course instructor. There are no make-up sessions for lab courses. If you are unable to attend due to an excused situation, you must contact **Dr. Kübra GÜRCAN BAYRAK (kubragurcan@eskisehir.edu.tr)** and provide the relevant documentation as proof.
11. For the face to face lessons, it is obligatory to **wear laboratory apron, safety glass, gloves and dust mask unless the lab instructor tells otherwise.** Students without aprons (lab coats) and masks will not be admitted to the laboratory.
12. The lab groups must be **present in the room/building** where the lab will take place (stated in the lab manual) **5 minutes before the lab** starts. Students are obliged **to learn the location of the labs** before the labs begin.
13. During the final week of the **Some Advanced Ceramics Lab Tour**, no quiz will be administered. However, **attendance is mandatory!** The topics covered in this week, which are **not included in the report content**, will be **part of the final exam.**
14. Lab manuals will be available **on the department web-sites**.
    https://matse.eskisehir.edu.tr/tr/Icerik/Detay/mlz331-2

## INSTRUCTION OF REPORT I

You are required **to submit a detailed formal report covering Exp-1, Exp-2, and Exp-3.** The report must follow the structure and the include the following sections:

### 1. TITLE PAGE:

It contains:

- Your full name
- Your student number
- Your group name

Ensure that this information is clearly visible.

### 2. ABSTRACT

This section should summarize the purpose and results of the experiments. The abstract should:

- Be concise and informative.
- Include the aims of the experiments and a brief summary of the key findings.
- Have a maximum length of 250 words.
- Avoid detailed explanations of methodology; focus on the overall outcomes and conclusions.

### 3. TABLE OF CONTENTS

Include a table of contents that outlines the different sections of the report with corresponding page numbers for easy navigation.

### 4. BACKROUND

This part should introduce the experiments, addressing:

- The rationale behind conducting these experiments
- How the experiments are related to each other
- Relevant literature that supports the experimental objectives
- It should include **references to reliable scientific sources (e.g., peer-reviewed journals, textbooks).** **Do not rely on general websites!**
- It should not exceed one page.

## 5. EXPERIMENTAL PROCEDURE

This section must:

- Begin with **a flow chart** that visually summarizes the experimental steps.
- Carefully describe the methods, materials, and equipment used in the experiments
- Provide details on the specific parameters used for the materials studied and the apparatus involved
- Aim for clarity and precision in describing the procedures so that they can be easily replicated by another researcher

## 6. RESULTS AND DISCUSSION

- Present the results obtained from the experiments, including relevant data, graphs, or tables
- Interpret the data and discuss its significance in relation to the experimental objectives
- Compare your findings to those in the literature, discussing any discrepancies or notable observations.

**<u>This section is crucial and should reflect your critical thinking and understanding of the experimental outcomes</u>**

## 7. CONCLUSIONS

Provide a concise summary of the key results and the overall significance of the experiments. The conclusion should:

- Recap the main findings without repeating too much detail
- Highlight the importance and implications of the experiments.

## 8. REFERENCES

This section is vital and must be handled with care. Your references should:

- Not solely consist of web pages; prioritize books, journal articles, and academic papers
- Follow a consistent citation style (e.g., APA, MLA, or your preferred style)
- Ensure all sources cited in the text are included here.

## INSTRUCTIONS FOR REPORT II

It will contain the answers to the questions at the end of the experiment manual, including 2 questions for each experiment. The report must follow the structure and the include the following sections:

### 1. TITLE PAGE:

It contains:

- Your full name
- Your student number
- Your group name

Ensure that this information is clearly visible.

### 2. ANSWERS THE QUESTIONS

**The relevant questions must be included in the report along with the question number and the corresponding answers.**

### 3. REFERENCES

Since **some of the questions will require literature research, your answers should be include references:**

This section is vital and must be handled with care. Your references should:

- Not solely consist of web pages; prioritize books, journal articles, and academic papers
- Follow a consistent citation style (e.g., APA, MLA, or your preferred style)
- Ensure all sources cited in the text are included here.

**Note: The EXPERIMENT MANUAL should not be used as a reference. Aim to search and cite reputable academic sources.**

# SCHEDULE

| Date of the Week | Laboratory Instructors | Experiment Name | Lab Locations |
|---|---|---|---|
| 01.10.2024 | Res. Assist. Dr. Kübra GÜRCAN BAYRAK | **Team Meeting** | |
| 07.10.2024-11.10.2024 | Res. Assist. Dr. Enes İbrahim DÜDEN | **Experiment♯1**<br>Raw Material Preparation and Particle Size Analysis | **MLZ 123**<br>**Ceramic Process Lab I**<br>**(Seramik Süreçler Lab I)** |
| 14.10.2024-18.10.2024 | Res. Assist. Ertuğrul İŞLEK | **Experiment ♯2**<br>Tile Production and Dry Pressing | **MLZ 123**<br>**Ceramic Process Lab I**<br>**(Seramik Süreçler Lab I)** |
| 21.10.2024-25.10.2024 | Res. Assist. Dr. Kübra GÜRCAN BAYRAK | **Experiment ♯3a**<br>Sintering of Ceramics<br>**Experiment♯3b**<br>Density and Porosimetry | **MLZ122**<br>**Advanced Ceramic Lab**<br>**(İleri Tek. Seramik. Lab)**<br>**MLZ 117**<br>**X-Rays Lab**<br>**(X-Işınları Lab)** |
| **Submission of First Report (28.10.2024-01.11.2024)** | | | |
| 04.11.2024-08.11.2024 | Res. Assist. Emine ERSEZER | **Experiment ♯4** | **MLZ 123**<br>**Ceramic Process Lab I**<br>**(Seramik Süreçler Lab I)** |
| 25.11.2024-29.11.2024 | Res. Assist. Dr. Levent KÖROĞLU<br>Res. Assist. Gülseda ŞENEL | **Experiment♯5a**<br>Particle Dispersion and Slip Casting - I | **MLZ 123**<br>**Ceramic Process Lab I**<br>**(Seramik Süreçler Lab I)** |
| 02.12.2024-06.12.2024 | Res. Assist. Dr. Levent KÖROĞLU<br>Res. Assist. Gülseda ŞENEL | **Experiment ♯5b**<br>Particle Dispersion and Slip Casting – II | **MLZ 123**<br>**Ceramic Process Lab I**<br>**(Seramik Süreçler Lab I)** |
| 09.12.2024-13.12.2024 | Res. Assist. Dr. Enes İbrahim DÜDEN<br>Res. Assist. Gülseda ŞENEL | **Some Advanced Ceramics Lab Tour** | - |
| **Submission of Final Report (16.12.2024-20.12.2024)** | | | |
| **MIDTERM EXAM (23-27.12.2024)** | | | |

## GROUPS

| | | | |
|---|---|---|---|
| **Monday** | **11:00 / 13.00** | **“Group A”** | **Res. Assist. Dr. Levent KÖROĞLU** |
| **Monday** | **16:00 / 18:00** | **“Group B”** | **Res. Assist. Dr. Enes İbrahim DÜDEN** |
| **Tuesday** | **16.00 / 18.00** | **“Group C”** | **Prof. Dr. Ferhat KARA** |
| **Wednesday** | **09.00 / 11.00** | **“Group D”** | **Prof. Dr. Alpagut KARA** |
| **Thursday** | **09.00 / 11.00** | **“Group E”** | **Res. Assist. Dr. Kübra GÜRCAN BAYRAK** |
| **Friday** | **09.00 / 11.00** | **“Group F”** | **Prof. Dr. Semra KURAMA** |

# GRADING TABLE

| Exam | Exam Type | Percentage of Exam |
|---|---|---|
| **SHORT EXAMS (QUIZS)** | **Exp#1, Exp#2, Exp#3, Exp#4, Exp#5,<br>Quizzes** | **25 %** |
| **II. MIDTERM** | **Exam** | **25 %** |
| **FINAL** | **REPORTS<br>First Report (30%)<br>Final Report (20%)** | **50%** |

# EXPERIMENT 1

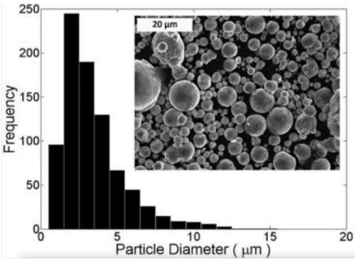
## RAW MATERIAL PREPARATION AND PARTICLE SIZE ANALYSIS

## 1. Objective of the experiment

- To learn industrial techniques for **size reduction and homogenization** of raw materials with *wet milling*
- To **measure particle** of ceramic powders with laser diffraction technique

## 2. What should you know before the experiment?

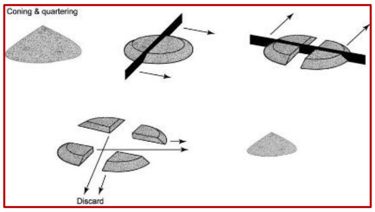
- **Sampling methods** and their usage (Alternative Shovel, Cone and Quartering, Splitting methods, Dividing by spatula)
- Particle **size reduction** methods (Crushing, grinding, milling)
- Milling types, equipments and parameters (wet milling, dry milling, gyratory mill etc, milling jar etc…)
- Common **particle size measurement** methods (Sieving, Laser Diffraction, BET, sedimentation etc.)
- Laser diffractometer principle
- BET principle
- Microscobic techniques (SEM, etc)



## 3. What will you learn during the experiment?

- How to **prepare** representative samples?
- How to carry out wet milling? What are the **important parameters?**
- What are the **differences** between the milling processes of traditional and advanced ceramics?
- What are the compositions of **wall, floor and porcelain** tile compositions?
- How to **determine** particle size distribution by laser diffraction methods?

# 4. Schematic view of experimental procedure



**Preparation of representative sample**

**Traditional ceramic powder wet milling in ball mill**

**Liter weight measurement by picnometer**

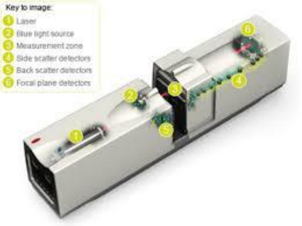
**Drying of the slurry in the oven**

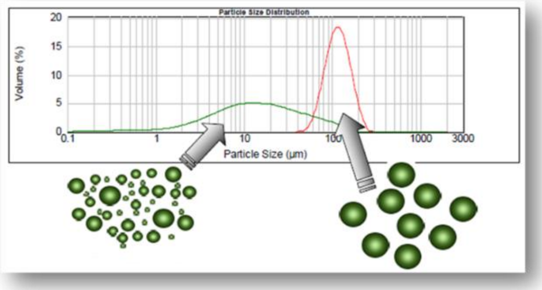
**Advanced ceramic powder wet milling in planetary mill**

**Transferring the slurry to the evaporation flask**

**Drying of the slurry with rotary evaporator**

**Particle Size analysis by using Laser Diffraction instrument**





# 5. Equipments and materials

- **For Representative Sample Preperation ;**

  Raw materials, ruler, shovel, splitting machine, spatule, sample container

- **For Wet milling of ceramics;**

  **For traditional ceramics:** Tile raw materials, balance, water, ball mill, $Al_2O_3$ jar and balls, picnometer, oven, laser diffraction instrument.

  **For advanced ceramics:** $Al_2O_3$ powder, balance, ethanol, $Si_3N_4$ jar and ball, evaporation flask, rotary evapotator, laser diffraction equipment

# 6. Important points / hints for the equipments and/or results obtained from the analyses

- **Why** particle size control is important and why particle size analysis should be performed
- Using complementary techniques (e.g, laser diffraction) for precise characterization of **particle size and size distribution**
- **Choosing** appropriate milling equipment and particle size analysis techniques of traditional and advanced ceramics
- Reasonable **interpretation** of the results

**HINT: The formulation of raw materials of tile compositions**

| Floor tile | Porcelain tile | Wall tile |
|---|---|---|
| Clay<br>30%wt | Kaolin<br>20%wt | Clay<br>50%wt |
| Kaolin<br>25%wt | Clay<br>10%wt | Kaolin<br>30%wt |
| Na-felspar<br>20%wt | Ukraine Clay<br>20%wt | Calcite<br>10%wt |
| Pegmatite<br>25%wt | Na-felspar<br>50%wt | Pegmatite<br>10%wt |

# MLZ331

# Report Sheet I

**Name & Surname:** **Group:**

**Number:**

## Experiment-1

### *Representative Sample Techniques*

1.……………………………………………

2.……………………………………………

3.……………………………………………

4.……………………………………………

### *Milling part of traditional ceramic*

**Materials Composition&Amount (gr)**

**1. …………………………………..**

**2. …………………………………..**

**3. …………………………………..**

**4. …………………………………..**

**Total amount of powder mixture …… (gr)**

**Deffloculant: ….... (% of the solid volume)**

**Solid:liquid ratio: ……………....**

**Ball to powder ratio: ……………**

**Jar&ball material: …….............**

**Extend of volume: ……………...**

**Milling speed: …………………(rpm)**

**Milling time:………………………**

**Liter weight of slurry: ……………….. (gr/lt)**

**Drying temperature: ……………………(°C)**

**Drying time: ………………………. (h)**

### *Milling part of advanced ceramic*

**Material: …………**

**Material amount: ………….. (gr)**

**Liquid type: …………………**

**Solid:liquid ratio: ……………....**

**Ball to powder ratio: ……………**

**Jar&ball material: …….............**

**Jar volume: ………………... (ml)**

**Extend of volume: ………………**

**Milling speed:………………...(rpm)**

**Milling time:………………………**

**Evaporation conditions:**

**Temperature: ………………… (°C)**

**Speed: …………………………. (rpm)**

**Media: ………………………….**

## Laser Diffraction Technique

**Sample: ……………….** **Refractive index: …………………....**

**Media: …………….......** **Absorption: ………………………….**

# EXPERIMENT 2

## TILE PRODUCTION AND DRY PRESSING

## 1. Objective of the experiment

- To learn particle size reduction of raw materials via **gyratory milling.**
- To determine **particle size distribution** of powders via sieve analysis.
- To show how to granulate traditional ceramic powders.
- To show how to shape wall tiles by **pressing.**
- To show the **pressure** and **thickness effect** on green density.

## 2. What should you know before the experiment?

- Milling types, equipments and parameters (wet milling, dry milling, gyratory mill etc, milling jar etc.)
- Common **particle size measurement** methods (Sieving, Laser Diffraction, BET, sedimentation etc.)
- **Granulation methods** of ceramic powders.
- What are the stages of pressing?
- What is the importance of powder characteristic?
- What are the parameters that should be considered during pressing?
- What are the defects that occurred during and after pressing?

## 3. What will you learn during the experiment?

- How to carry out **gyratory milling**?
- How to determine particle size distribution by **sieving**?
- How to **granulate** traditional ceramic powders?
- How to form ceramic powders by using **dry pressing**?
- How the **pressure** and **thickness** affects the green density?
- How can the defects occur during and after pressing?
- How to control the **compaction defects**?

# 4. Schematic view of experimental procedure

*Gyrotary milling of traditional ceramic powders prepared in the first experiment for wall tile production*

⇩

*Particle size analysis via sieving*

⇩

*Granulation of powders*

⇩

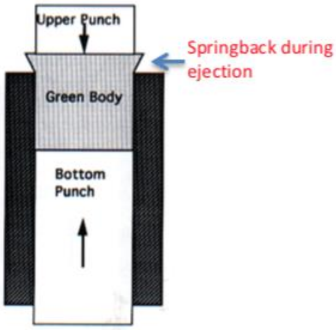
*Weighing the powder, filling the die and pressing under designated pressure*

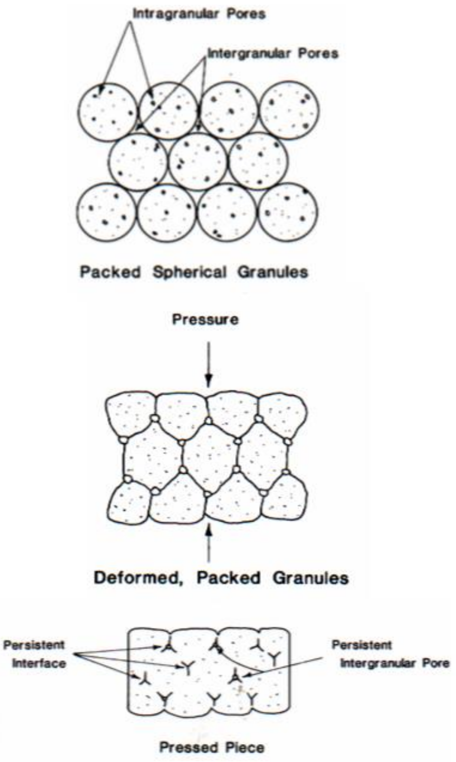
⇩

*Ejecting the sample out of the die*

⇩

*Measure and record weight and dimension of the sample*





## 5. Equipments and materials

- Gyratory Mill,
- Sieve Equipment,
- Ceramic Powder (Granulated),
- Pressing Dies and Mechanical Press.

## 6. Important points / hints for the equipments and/or results obtained from the analyses

- **Why** particle size control is important and why particle size analysis should be performed.
- Good powder flow is essential for reproducible volumetric filling, a uniform density of the fill and a rapid pressing rate.
- Hard granule difficult to change shape, causing residual pore, thus lowering product strength.
- The compact must survive ejection and handling without failure and should be free of defects.
- Using lubricant during pressing is important to be aware of frictional forces.
- To minimize defect formation, some pressure is kept during ejection process
- Air problem can be minimized by de-airing before compression.

# Report Sheet 2

**Name & Surname:** **Group:**

**Number:**

## Experiment-2

## 1. Measurements

### 1.1 Sieve Analysis

| Sieve Size (um) | Weight Retained (g.) | Cumulative Weight Retained (g.) | Cumulative Retained (%) | Cumulative Passing (%) |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

*Graph of sieve analysis will be included in first formal report (Due Date: (28 October-1 November)

### 1.2 Pressing Conditions, Dimensions and Weight of Green Body

| Pressure (MPa/Bar) | Thickness (mm) | Weight (g) |
|---|---|---|
| | | |
| | | |
| | | |

| Radius (cm.) | Thickness (cm.) | Weight (g.) |
|---|---|---|
| | | |
| | | |
| | | |
| | | |

**Average** →

| Radius (cm.) | Thickness (cm.) | Weight (g.) |
|---|---|---|
| | | |
| | | |
| | | |
| | | |

**Average** →

# EXPERIMENT 3

## Sintering of Ceramics

## 1. Objective of the Experiment

- To learn **conventional and non-conventional** sintering process of ceramic materials.
- To evaluate sintering graphs.
- To measure **the density of sintered ceramic** bodies.

## 2. What should you know before the experiment?

- What is sintering?
- What are **Solid State Sintering, Liquid Phase Sintering, Viscous Flow Sintering and Presure Assisted Sintering?**
- What **types of furnaces** are used for sintering ceramics?
- What are the basic components of a furnace?
- What are the density and porosity?
- What is **Archimede’s** principle?

## 3. What will you learn during the experiment?

- How to estimate the sintering profile of a ceramic body.
- How to sinter ceramics powders to a dense body.
- The sintering mechanisms and material transport mechanisms during sintering processes.
- How sintering parameters affect properties of the final product.
- How to measure the density of sintering product.
- How to calculate the density of porous materials.

# 4. Schematic view of experimental procedure

## Conventional sintering

*Placement of the dry-pressed samples prepared in Exp-2 into the muffle furnace*

⇩

*Sintering*

⇩

*Recording of the sintered samples dimensions*

⇩

*Evaluation of the data after sintering*

## Non-Conventional sintering

*Explanation of the SPS furnace components*

⇩

*Filling the die with prepared $Al_2O_3$ powder in Exp-1&2*

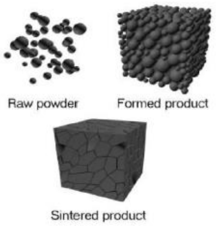
⇩

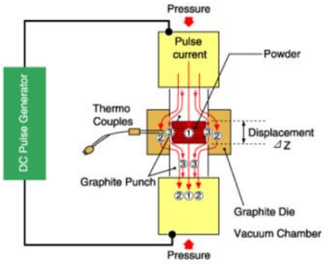
*Sintering*

⇩

*Evaluation of the data obtained from the SPS*

*Density measurement*





# Density measurement

Weight the dry sample

⇩

Put in the distilled water

⇩

Soak in boiling water for 4 hours

⇩

Wait until the water reaches RT

⇩

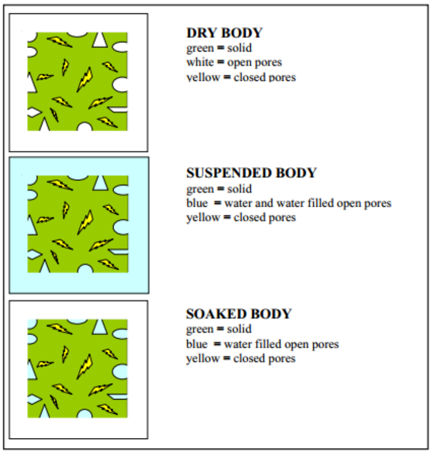
Weight the sample in water ($W_w$)

⇩

Clean the sample and weighing air ($W_A$)

⇩

Calculation



- **Bulk Density** $(BD) = \frac{W_{dry}}{W_A - W_w} \; x \; \rho_{water}$
- **Water absorbtion** $(\%WA) = \frac{W_A - W_{dry}}{W_{dry}} \; x \; 100$
- **Apparent Porosity** $(\%AP) = \frac{W_A - W_{dry}}{W_A - W_w} \; x \; 100$
- **Apparent Solid Density** $(ASD) = \frac{W_{dry}}{W_{dry} - W_w}$

## 5. Equipment and Materials

- **For non-conventional sintering:** the dry-pressed samples prepared in Exp-2, muffle furnace, oven glove, tongs, caliper.

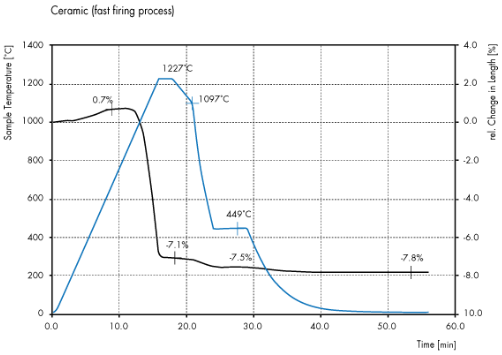
- **For conventional sintering:** the milled and sieved samples prepared in Exp-1 and Exp-2, graphite tools (paper, die, punch, blanket etc) for sintering, spark plasma sintering furnace.

- **For density measurement:** Presicion scale, Archimedes density equipment, forceps, napkin.

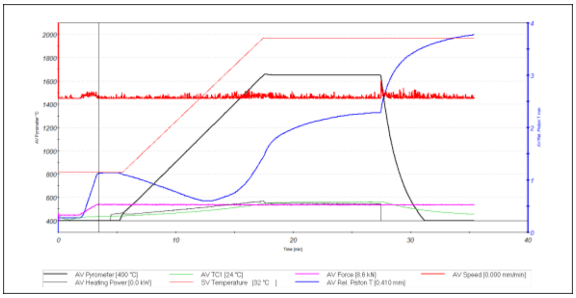
## 6. Important points/hints for the equipments and/or results obtained from the analyses.

- Sintering process parameters
- Correct preparation of the die in non-conventional sintering
- Reasonable interpretation of the sintering graphs
- Density evaluation.

***HINT:*** Representation of sintering graphics:



Dilatometer graph of conventional sintered ceramic sample



SPS graph of non-conventional sintered ceramic sample

# MLZ331

# Report Sheet 3

**Name & Surname:** **Group:**

**Number:**

## Experiment-3

### *Sintering of Dry Pressed Samples*

**Sintering parameters:**

**Temperature: …………………….**

**Time: ……………………………….**

**Atmosphere……………………….**

**% Shrinkage of samples:**

**For sample 1:**

**Volume before sintering: ………(cm$^3$)**

**(From Exp-2)**

**Volume After sintering: …………… (cm$^3$)**

**%shrikage: …………………**

**For sample 2:**

**Volume before sintering: ………(cm$^3$)**

**(From Exp-2)**

**Volume After sintering: …………… (cm$^3$)**

**%shrikage: …………………**

**Water absorption of samples**

**For sample 1:**

**$W_w$=…….............. / $W_a$=……………………**

**Water absorption (%) : …………………...**

**For sample 2:**

**$W_w$=…….............. / $W_a$=……………………**

**Water absorption (%) : …………………...**

### *Sintering of advanced ceramics*

**Preperation of sample:**

**Die material: ………………………..**

**Die diameter (mm): ………………..**

**Sample: ……………………………...**

**Sample amount (gr): ……………....**

**Sintering parameters:**

**Temperature (°C): …………………….**

**Pressure (kN) ………………………….**

**Heating rate (°C/min): ……………......**

**Dwell time (min): ……………………...**

**Total process time (min) …………......**

**Atmosphere: …………………………..**

**Density of Sample:**

**$W_{dry}$ ……………....... (gr)**

**$W_w$: ……………....... (gr)**

**$W_a$: ……………....... (gr)**

**Bulk density (g/cm$^3$): ........................**

**Apparent porosity (%): ……………….**

**Apparent solid density: ………………**

# EXPERIMENT 4

## GLAZING

## 1. Objective of the Experiment

- To show how to apply glaze and explain the most important parameters in terms of obtaining decent glazes.

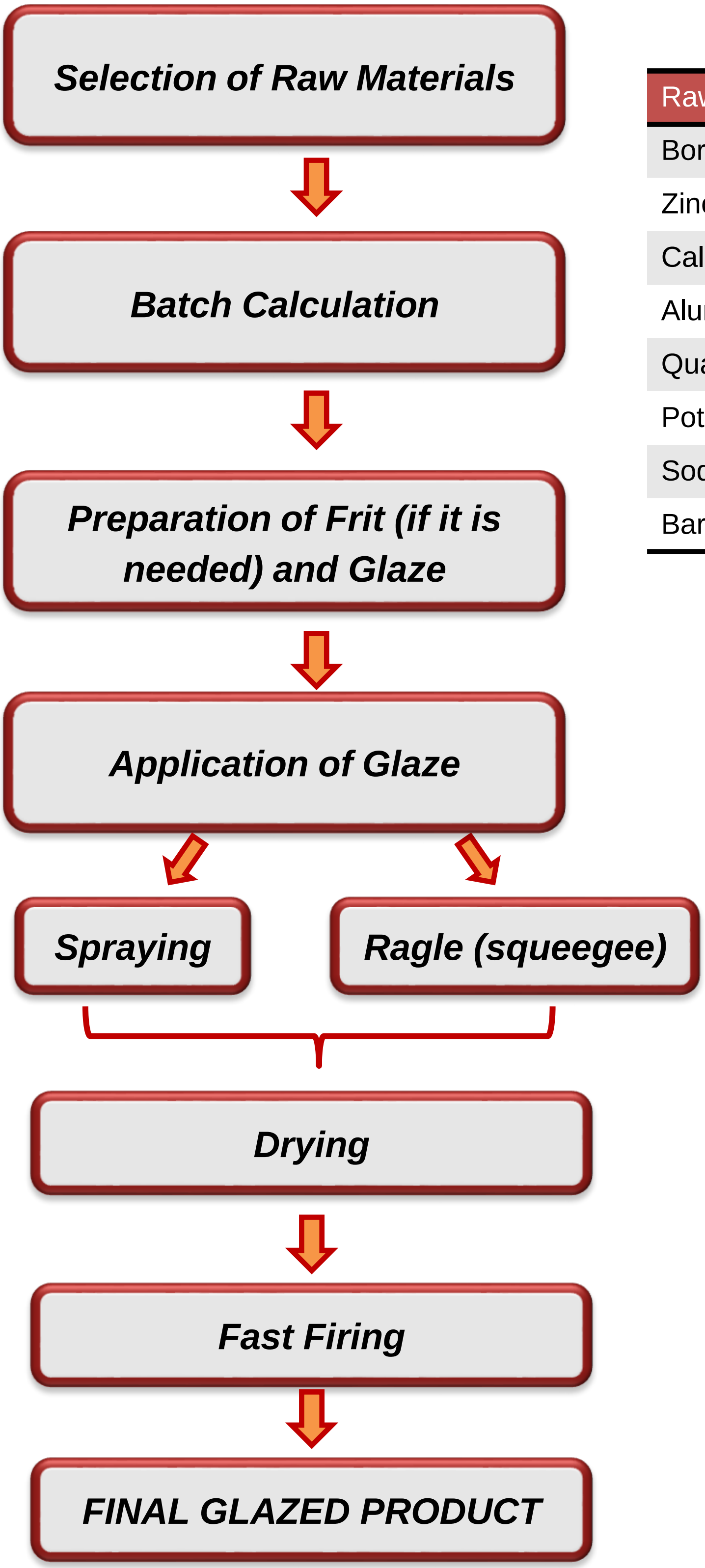
## 2. What should you know before the experiment?

- What is **glaze**?
- Why are glaze coatings applied to products?
- What are the most important parameters in terms of obtaining decent glazes?
- What are the different methods of glaze application?
- Why is the rheology of the glaze important?
- What does **deflocculant provide**?

## 3. What will you learn during the experiment?

- Why **is frit used** in glazes?
- What are the differences between **fritted and unfritted glaze**?
- What properties are supplied to products by applying glaze?
- What are the types of glazes?
- Which raw materials are used for preparing glaze and what are their functions?
- How do we apply glaze on the products?
- Why do we use CMC and STPP?
- How do we determine the firing temperature for a glaze?
- What are the **common glaze defects**?

# 4. Schematic view of experimental procedure



Selection of Raw Materials

Batch Calculation

Preparation of Frit (if it is needed) and Glaze

Application of Glaze

Spraying

Ragle (squeegee)

Drying

Fast Firing

FINAL GLAZED PRODUCT

**Glaze Recipe**

| Raw Materials | Weight % |
|---|---|
| Boric acid | 5.46 |
| Zinc oxide | 6.54 |
| Calcite | 12.64 |
| Alumina | 16.44 |
| Quartz | 34.42 |
| Potassium nitrate | 3.45 |
| Sodium feldspar | 15.35 |
| Barium carbonate | 5.71 |

## 5. Equipments and materials

Slurry of glaze composition (commercially obtained)

Marsh Cone apparatus

Spraying and ragle apparatus

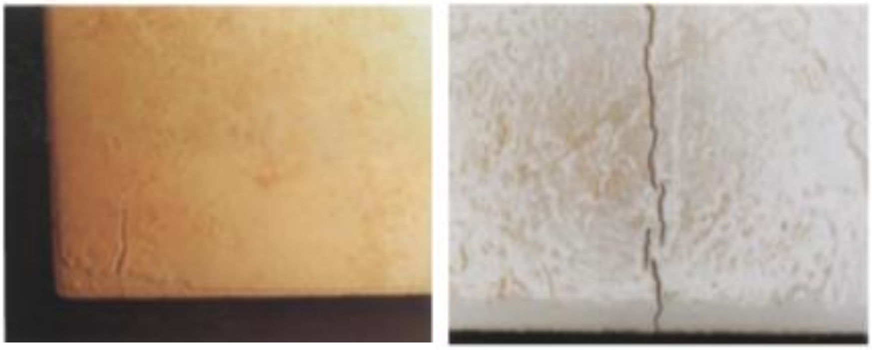
Drying oven

Fast firing furnace

## 6. Important points / hints for the equipments and/or results obtained from the analyses

- Criteria for materials' selection
- Milling parameters
- Application techniques
- Firing
- Body-glaze interactions

**Hints:** One of the glaze defect: **Crazing**



# MLZ331

# Report Sheet-4

**Name & Surname:** **Group:**

**Number:**

## Experiment-4

### *Glaze Application Techniques*

1) ………………………………………………

2) ………………………………………………

### *Glaze Preparation and Application*

**Glaze Composition&Amount (wt%)**

1. …………………………………..
2. …………………………………..
3. …………………………………..
4. …………………………………..
5. ………………………………….
6. ………………………………….
7. …………………………………....
8. ………………………………...

**Type of Body and Glaze:** ……………………………………………..

**Density of glaze:** …………………………………….……………….... (gr/lt)

**Viscosity measurement method:** ………………………………..…..

**Flow time of glaze during viscosity measurement:** ……….……… (s)

**Drying temperature:** …………………………….(°C)

**Drying time:** ……………….…………….……… (h)

**Firing temperature :** ………………..…………….(°C)

**Firing time:** …………………………………..……. (h)

# EXPERIMENT 5

## PARTICLE DISPERSION AND SLIP CASTING

## 1. Objective of the Experiment

- To gain main knowledge on **particle dispersion in ceramics and rheology of materials.**
- To **prepare sanitaryware slips** and **determine the casting properties** depending on the flocculation of slip.

## 2. What should you know before the experiment?

- Definitions of **viscosity, thixotropy, and Brownian motion.**
- **Newtonian behavior and Non-Newtonian behaviors**, such as dilatant (shear thickening), pseudoplastic (shear thinning), and Bingham plastic.
- Definitions of **electrical double layer, zeta potential, and slipping plane.**
- **The types of stabilization**; electrostatic, steric and electrosteric stabilization.
- The **microstructure of kaolinite particles** and **charge formation** on their basal planes **upon ionic dissolution**.
- The effect of **deflocculant** on kaolinite particles.
- The effect of **counter ions' concentration on double layer thickness.**
- The properties of **gypsum mold and capillary effect.**

## 3. What will you learn during the experiment?

- The **stabilization** of a ceramic suspension (slurry) and **thixotropic behavior.**
- The characterization of the **degree of stabilization**.
- The understanding **over deflocculation** phenomenon.
- **Slip casting** process of ceramic bodies.
- The effect of **viscosity-deflocculant content on casting rate** of green body**.**

# 4. Background

The coefficient of **viscosity**, η (Pa.s) indicates the resistance to flow due to internal friction between the molecules of the liquid. A shear rate, γ (1/s) is required to initiate and maintain laminar flow in a sample liquid. When a shear stress, τ (Pa) is linearly dependent on the velocity gradient (shear rate), liquid shows **Newtonian behavior**.

$\tau = \eta \,.\, \gamma$ ; where τ is shear stress (Pa); η is viscosity (Pa.s); and γ is shear rate (1/s).

**Non-Newtonian** materials provide a nonlinear dependence of shear stress on shear rate. If the viscosity decreases with increasing shear rate, behavior is said to be **pseudoplastic (shear thinning)**. In contrast, flocculated slurries show **dilatant (shear thickening)** behavior, where viscosity increases with an increase in the shear rate. Slurries containing a linkage of bonded molecules and particles require a yield stress to initiate flow. It is known as **Bingham plastic behavior**.

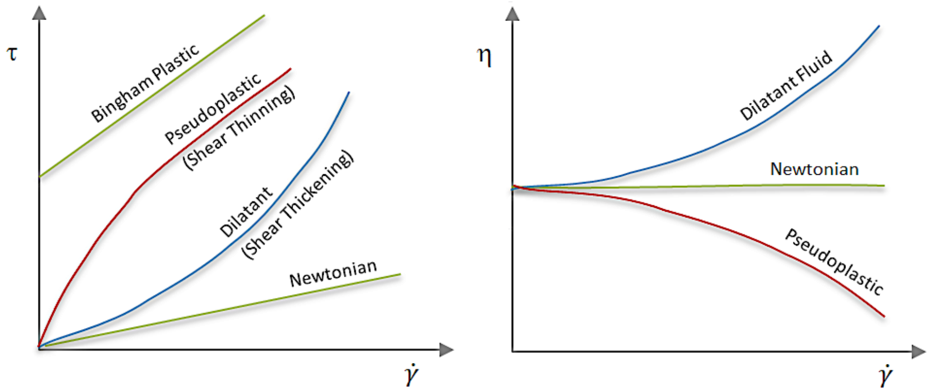


**Fig. 1.** Schematic a) flow and b) viscosity curves of Newtonian and non-Newtonian materials

**Thixotropy** is a time-dependent shear thinning property. Some non-Newtonian pseudoplastic fluids show a time-dependent change in viscosity; the longer the fluid undergoes shear stress, the lower its viscosity.

Colloidal particles in a solution are continuously bombarded by the molecules of the suspension medium on all sides. The impacts are however not equal in every direction. As a result, the colloidal particles show random or zig-zag movements, which is called **Brownian motion**.

Charged particles in a suspension will respond to an imposed potential difference. During flow, a slipping plane must occur somewhere in the electrical double layer. The potential at the slippage plane is called the **zeta potential (ζ-potential).**

In a suspension, the surface of a charged particle is balanced by an equal number of oppositely charged counter ions in solutions. The surface charge on a particle and counter ion charge form an electrically neutral electrical double layer. Through the moving of a colloidal particle in suspension, a layer of the surrounding liquid remains attached to the particle. The boundary of this layer is known as the **slipping plane** (shear plane). **Zeta potential (ζ-potential)** is the value of the electric potential at the slipping plane.

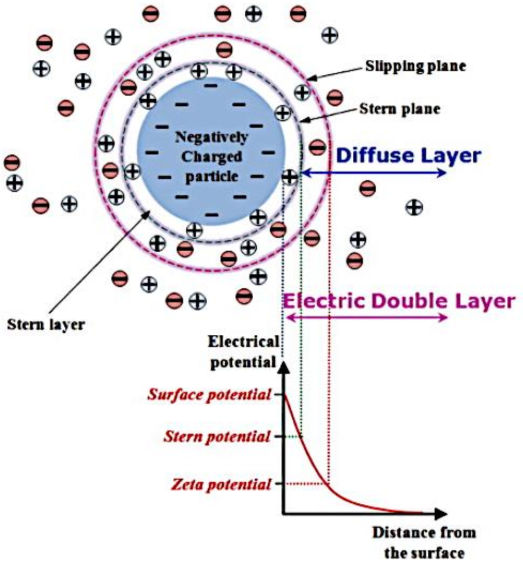


**Fig. 2.** Diagram of electric double-layer

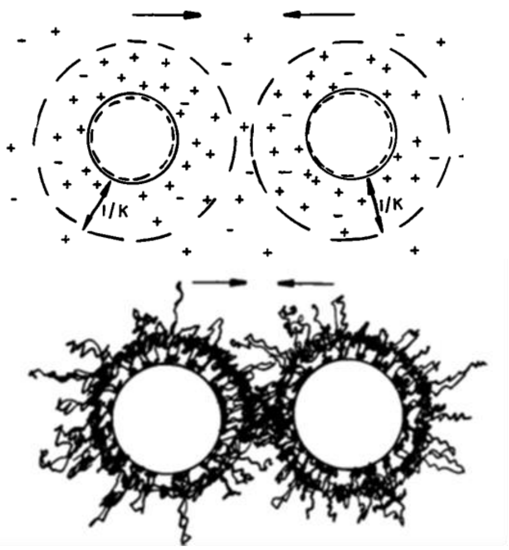


**Fig. 3.** Schematic representation of electrostatic and steric stabilization

The **stabilization** of a ceramic suspension (slurry) refers that ceramic particles in the liquid continue to exist as individual units. The dispersion stability is provided by preventing the agglomeration of particles. **Electrostatic stabilization** is attained by electrical charges on the surfaces of particles, while **steric stabilization** is imparted by macromolecules attached to the surfaces of particles. **Electrosteric stabilization** refers to the combinations of electrostatic and steric stabilization.

A **deflocculant** (e.g. sodium silicate) is mostly used as additive to achieve electrostatic stability of ceramic suspensions by increasing the repulsive forces among ceramic (kaolin) particles. In the case of sodium silicate, positively charged $Na^+$ ions are attracted by the **basal planes of kaolin particles,** which become **negatively charged** upon dispersion when adsorbed alkali ions are liberated. The **concentration** of positive **counter ions** on the surface of charged kaolin particles determines **zeta potential, double layer thickness, and stability of ceramic suspension**.

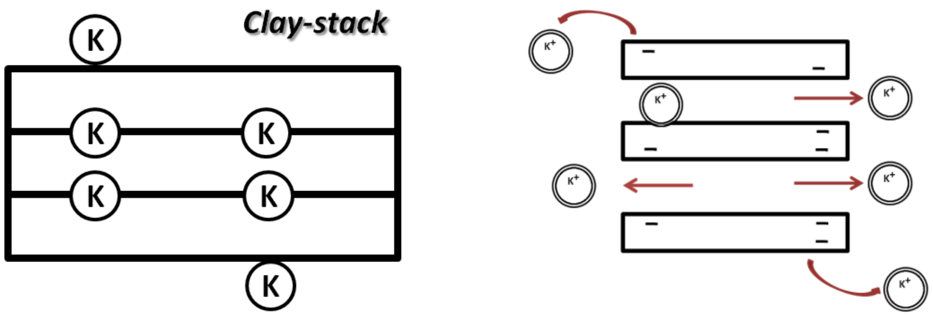
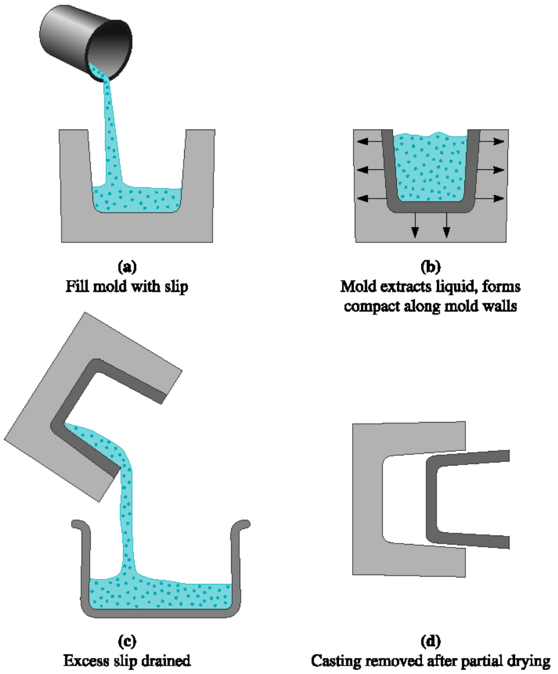


**Fig. 4.** Kaolin particles a) before and b) after dispersion

The viscosity of a ceramic suspension can be monitored by the deflocculant concentration. The degree of stabilization significantly affects **slip rheology and casting rate (thickness of cast).** During slip casting, **porous gypsum molds** extracts the liquid of sanitaryware slip through **capillary action.** Casting rate is controlled by **permeability** of the cake. **High casting rate** is required for **a sanitaryware cast** because some retained water in the cast provides **plasticity.**



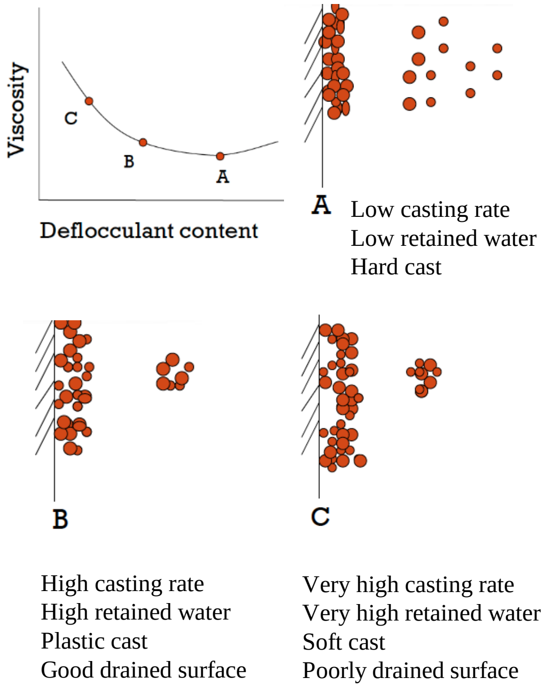


**Fig. 5.** Slip casting process

**Fig.6** Effect of slip structure on casting

*Reed, J.S. (1995). Principles of Ceramic Processing (2nd Ed.) New York: John Wiley & Sons, Inc.*
*Lee, J.K., Ko, J., and Kim Y.S. (2017). Minerals, 7 (165), 1-11.*
*Jain S.K. and Shailesh K. Jain. (1986). Conceptual Chemistry Volume-I For Class XII, New Delhi: S. Chand Company Pvt. Ltd.*
*Napper, D.H. (1983). Polymeric Stabilization of Colloidal Dispersions. London: Academic Press.*
*Park, S.-J., and Seo, M.-K. (2011). Intermolecular Force. S. J. Park, M. K. Seo (Ed.), Interface Science and Composites (pp. 1–57). Amsterdam: Academic Press.*

## 5. Schematic view of experimental procedure

**Weigh up raw materials**

⇩

**Slurry preparation**
**(Two different slurries are prepared by each lab. group)**

⇩

**Adjusting viscosity by deflocculant content**
**(One slurry is over-deflocculated in purpose)**

⟺

**Viscosity measurements**
**(stir slurry for 3 min and then record the viscosity value on 15th second)**

⇩

**Slip casting using three gypsum molds**
**(Each slurry is partially dried for 5, 10, and 20 min.)**

⇩

**Cast thickness measurements**
**(Three measurements are done for each slip cast)**

## 6. Equipments and materials

**For slurry preparation (for each slurry);**
500 g kaolin, distilled water (solid/liquid ratio will be explained), sodium silicate ($Na_2SiO_3$) as deflocculant, 2 plastic beakers (500 and 1000 ml), 1 mixer (stirrer) with a mixer tip, 2 balances, Brookfield viscosimeter with a spindle, spatula, knife, plastic pasteur pipette, aluminum foil, paper and tape.

**For slip casting (for each slurry);**
3 gypsum molds, digital caliper, and 2 plastic beakers.

# 7. Important points / hints for the equipments and/or results obtained from the analyses

- **During the experiment and reporting period**, consider the relationship among the terms: non-Newtonian behaviors including time dependent one, counter ion concentration, zeta potential, electrical double layer thickness, slip structure, casting rate, and drying time.
- **During the experiment,** don't forget to write down the amount of deflocculant used for each time; you will add a small amount of deflocculant into slurry and repeat it several times (about 20 times).
- **While drawing viscosity - cumulative deflocculant content graph**, don't forget to label y-axis viscosity and be carefull about the units (cP and wt%). The first deflocculant content value on graph indicates the total deflocculant amount used for the preparation of slurry. Deflocculant content should be in unit of wt% (solid-solid ratio): total amount of deflocculant (X g) / total amount of kaolin (500 g). Then, add the new deflocculant content values on graph (it increases cumulatively) considering the step of "adjusting viscosity by deflocculant content".
- **While drawing wall thickness - drying time graph**, don't forget to label y-axis wall thickness and be carefull about the units (mm and min).
- **While comparing the viscosity - wall thickness values at 10th minute**, two different slurries are prepared by each lab. group. Hence, you are obtained two different casts (green boddies) by slip casting after 10 min drying. Each sub-group will share the data with other one, and you should compare wall thickness values measured after 10 min drying. Also, be carefull about the units (cP mm and min).

# Report Sheet-5

**Name & Surname:** **Group:**

**Number:**

## Experiment-5

### *Glaze Application Techniques*

**1) ……………………………………………….**

**2) ……………………………………………….**

### *Glaze Preparation and Application*

**Glaze Composition&Amount (wt%)**

**1. …………………………………..**
**2. …………………………………..**
**3. …………………………………..**
**4. …………………………………..**
**5. ………………………………….**
**6. ………………………………….**
**7. ………………………………....**
**8. ……………………………….…**

**Type of Body and Glaze: ……………………………………………..**

**Density of glaze: ……………………………….……………….... (gr/lt)**

**Viscosity measurement method: ……………………………..…..**

**Flow time of glaze during viscosity measurement: ……….……… (s)**

**Drying temperature: …………………………….(°C)**

**Drying time: ………………..…………….……… (h)**

**Firing temperature : ………………..…………….(°C)**

**Firing time: ……………………………….……. (h)**

### Table 1. Deflocculant content used during the slurry preparation step

| | Initial | Final | Difference (used defloc. amount) (g) |
|---|---|---|---|
| 1. | | | |
| 2. | | | |
| 3. | | | |
| 4. | | | |
| 5. | | | |
| 6. | | | |
| 7. | | | |
| 8. | | | |
| 9. | | | |
| 10. | | | |
| 11. | | | |
| 12. | | | |
| 13. | | | |
| 14. | | | |
| 15. | | | |
| 16. | | | |
| 17. | | | |
| 18. | | | |
| 19. | | | |
| 20. | | | |
| 21. | | | |
| 22. | | | |
| 23. | | | |
| 24. | | | |
| 25. | | | |
| **Total deflocculant amount (g):** | | | |
| **Total deflocculant content (solid:solid mass ratio) (wt%)** | | | |

### Table 2. Deflocculant content used for adjusting viscosity step

| | Initial | Final | Diff. (g) | Diff. (wt%) |
|---|---|---|---|---|
| 1. | | | | |
| 2. | | | | |
| 3. | | | | |
| 4. | | | | |
| 5. | | | | |
| 6. | | | | |
| 7. | | | | |
| 8. | | | | |
| 9. | | | | |
| 10 | | | | |

### Table 3. Viscosity and cum. defloc. content

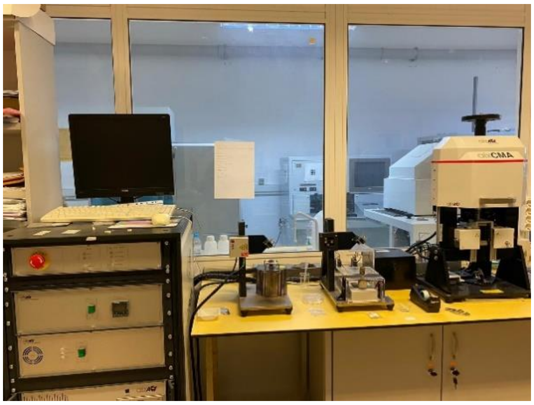
| Viscosity (cP) | Cumulative Deflocculant content (wt%) |
|---|---|
| *(after stirring)* | |
| | |
| | |
| | |
| | |
| | |
| | |
| | |
| | |
| (final viscosity) | |

(+)

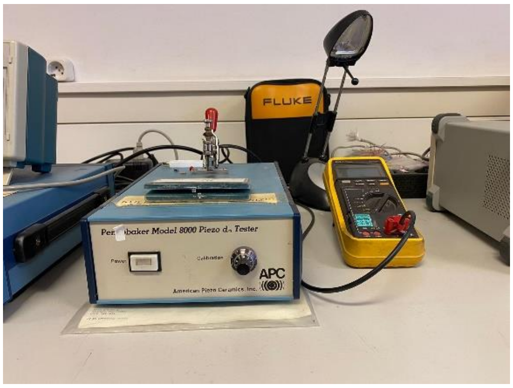
### Table 4. Drying periods and wall thicknesses

| Drying Period (min) | 1st | 2nd | 3rd | Avg. Wall Thickness of Cast (mm) |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |

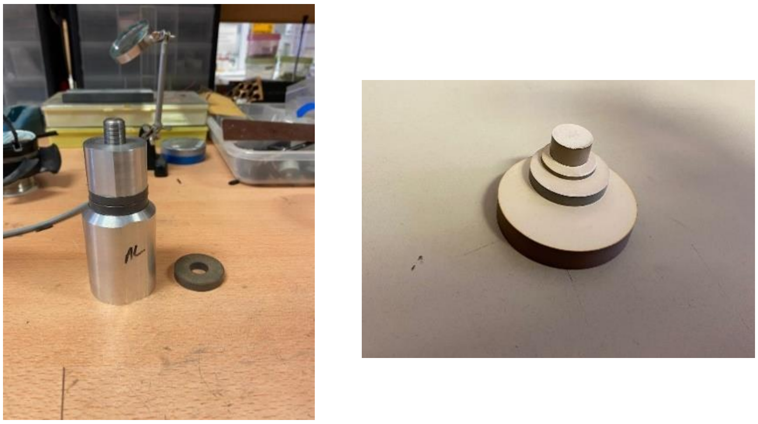
## Equipment within the Electorceramic Materials Laboratory



*Ferroelectric Property Measurement Device*

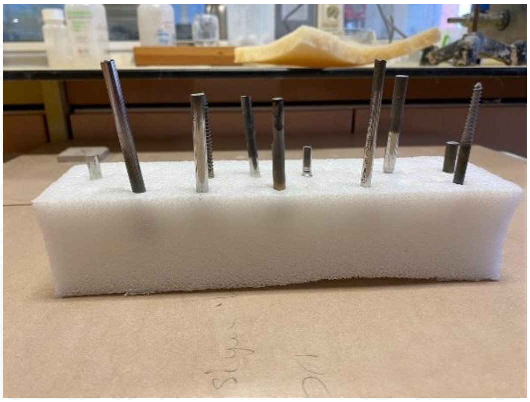


*Piezo d33 Tester*



*Piezoceramic specimens*

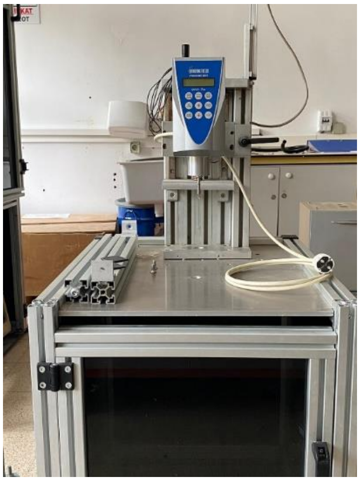
## Biomaterials



*Coated Implant Samples*

*Antimicrobial Powder*

## Equipment within the Glass Laboratory



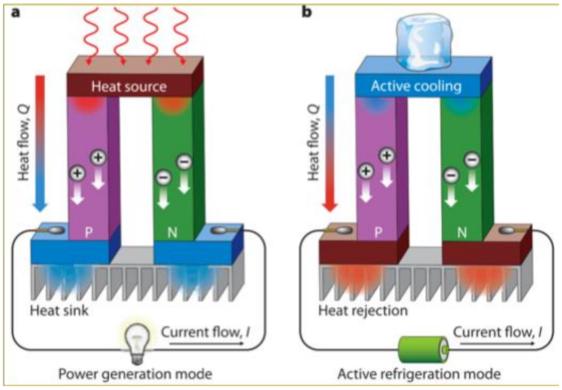
*Brookfield Viscometer*

*Glass fiber*

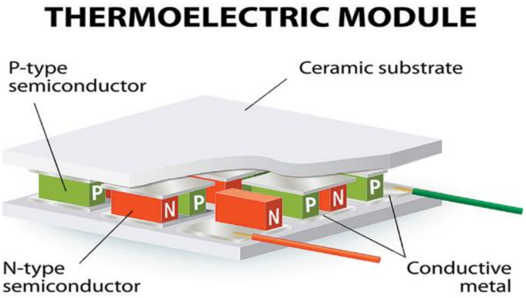
*Glass Stress Concentration Analyzer*

*Fiber Drawing Unit*

## Equipment within the Thermoelectric Laboratory



*Working Principle*



*Thermoelectric Characterization Device*

# QUESTIONS

## EXP-1

**1.** How does particle size reduction during wet milling affect the properties of the final ceramic product, and why is it important to achieve a uniform particle size distribution?

**2.** Discuss the advantages and disadvantages of different particle size and shape measurement techniques.

## EXP-2

**3.** How do granulation and the applied pressure during dry pressing influence the green density and overall mechanical properties of the ceramic tile?

**4.** What are the potential defects that can occur during the dry pressing process, and how can these be minimized?

## EXP-3

**5.** How does the shrinkage of a ceramic sample during sintering relate to the densification process? Can we always assume that more shrinkage results in a denser material? Why or why not?

**6.** What are the primary factors that influence the densification process during sintering, and how do they affect the quality of the final ceramic product?

Good Luck ☺

## EXP-4

**7.** How do the rheological properties, including viscosity, of the glaze affect the application process and the final quality of the glazed product? What additives are used to adjust these properties, and what are their specific purposes?

**8.** How does improper glaze application lead to common glaze defects such as pinholing or crazing, and how can these defects be minimized?

## EXP-5

**9.a.** During an experiment, you prepared a slurry in the first week and allowed it to rest for a week. In the second week, you measured the viscosity of the rested slurry and observed a high viscosity value. After stirring the slurry, you found a much lower viscosity. What could be the reasons for these observations?

**b.**You draw a viscosity - deflocculant content graph during experiment. One sub-group observed the increase of viscosity with deflocculant content after a minimum viscosity value was achieved. What is the reason of this phenomenon?

**10.** Each sub-group prepared a slurry during experiment and so you obtained two different casts by slip casting after 10 min drying. Why did you measured different wall thickness values for these casts, although they were dried for a same period of time?

Good Luck ☺