Eskisehir Technical University
Materials Science and Engineering Department

MLZ 331

Materials Processing Laboratory-I

2025 – 2026 FALL

Course Instructors

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Assist Prof. Dr. H.Boğaç POYRAZ
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Laboratory Instructors

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Res. Assist. Emine ERSEZER
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Res. Assist. Barış ÇİFTÇİ

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. For the face-to-face lessons, it is obligatory to **wear laboratory apron.** Students without aprons (lab coats) will not be admitted to the laboratory.
3. 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.
4. **At the beginning of experiments**, there will be **a quiz composed of 2-3 questions.**
5. **The final grade of course will be based on two components: (i) the completion of report sheets for each experiment, including answers to the end-of-experiment questions and (ii) one formal report to be submitted during the term.**
   - (i) Each experiment includes dedicated sections **in the report sheets**, which must be completed synchronously during the laboratory session. The report sheets, **together with the end-of-experiment questions,** must be submitted into the **designated boxes** on the dates announced in the schedule below.
   - (ii) Detailed instructions for preparing the formal report are provided under the section titled ***"11. INSTRUCTIONS FOR FORMAL REPORT."*** A two-week period is allocated for the preparation of this report due to its comprehensive nature. Students **are strongly advised not to postpone the work until the final days and must follow the instructions provided below. Formal Report** 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 **50339074 class ID and MLZ331 enrolment key**. Files should be uploaded by the name format of **GroupA_12345678910_AhmetYılmaz**. The formal report must be submitted **26.12.2025, 18:00** which is the last day of the two-week submission period.

6. Reports must be handed in on time; **otherwise 10% will be deducted from the mark for each day late.**

7. 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.**
8. 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.
9. Lab manuals will be available on the department web-site: https://matse.eskisehir.edu.tr/

10. The course grade will be based on the following:

GRADING TABLE

Exam	Exam Type	Percentage of Exam
SHORT EXAMS (QUIZS)	**5 QUIZS (Exp#1, Exp#2, Exp#3, Exp#4, Exp#5)**	**25 %**
MIDTERM	**Exam**	**25 %**
FINAL	1. **5 REPORTS (Exp#1, Exp#2, Exp#3, Exp#4, Exp#5 (20%)** 2. **Formal Final Report (30%)**	**50%**

11. INSTRUCTION OF FORMAL REPORT

You are required to submit **a detailed formal report covering all experiments during the term.** 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 300 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:

- Briefly explain the purpose and rationale of the experiments.
- Indicate the connection between the experiments in a few sentences.
- Include short references from reliable academic 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:

- Include **a flow chart** that visually **summarizes all experiments.**
- For each experiment, clearly state the **equipment used** and the **critical parameters**

Note: Report sheets will serve as the main source for this section.

6. RESULTS AND DISCUSSION

- Present the **key results** from the experiments; use tables or graphs when necessary.
- 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.

This section is crucial and should reflect your critical thinking and understanding of the experimental outcomes

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.

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	Report Submission
02.10.2025	Res. Assist. Dr. Kübra GÜRCAN BAYRAK	**Team Meeting**	**M4**	-
06.10.2025- 10.10.2025	Res. Assist. Dr. Kübra GÜRCAN BAYRAK	**Experiment♯1** Raw Material Preparation and Particle Size Analysis	**MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)**	13.10.2025- 17.10.2025
13.10.2025- 17.10.2025	Res. Assist. Barış ÇİFTÇİ Res. Assist. Emine ERSEZER	**Experiment ♯2a** Tile Production and Dry Pressing **Experiment ♯2b** Glazing	**MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)**	20.10.2025- 24.10.2025
20.10.2025- 24.10.2025	Res. Assist. Ertuğrul İŞLEK	**Experiment ♯3a** Sintering of Ceramics **Experiment♯3b** Density and Porosimetry	**MLZ122 Advanced Ceramic Lab (İleri Tek. Seramik. Lab) MLZ 117 X-Rays Lab (X-Işınları Lab)**	03.11.2025- 07.11.2025
REPUBLIC DAY HOLIDAY WEEK (27.10.2025-31.10.2025)				
03.11.2025- 07.11.2025	Res. Assist. Gülseda ŞENEL	**Experiment ♯4** Smart Ceramic Materials & Characterisation	**MLZ232 Electroceramic Lab MLZ233Advanced Tech. Lab MLZ121-2 Thermoelectric Lab**	24.11.2025 28.11.2025
MIDTERM WEEK (08.11.2025-23.11.2025)				
24.11.2025 28.11.2025	Res. Assist. Dr. Levent KÖROĞLU Res. Assist. Gülseda ŞENEL Res. Assist. Dr. Enes İbrahim DÜDEN	**Experiment ♯5a** Particle Dispersion and Slip Casting – I	**MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)**	-
01.12.2025- 05.12.2025	Res. Assist. Dr. Levent KÖROĞLU Res. Assist. Gülseda ŞENEL Res. Assist. Dr. Enes İbrahim DÜDEN	**Experiment ♯5b** Particle Dispersion and Slip Casting – II	**MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)**	08.12.2025- 12.12.2025
Submission of Final Report (15.12.2025-26.12.2025)				
EXAM (29.12.2025-31.12.2025)				

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”**	**Assist Prof. Dr. H. Boğaç POYRAZ**
Thursday	**14.00 / 16.00**	**“Group E”**	**Assist Prof. Dr. Mert GÜL**
Friday	**16.00 / 18.00**	**“Group F”**	**Res. Assist. Dr. Kübra GÜRCAN BAYRAK**

EXPERIMENT 1

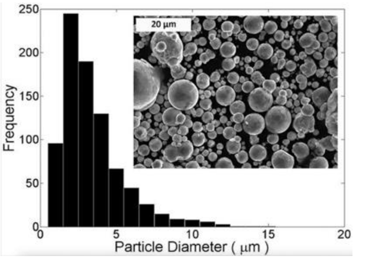
RAW MATERIAL PREPARATION AND PARTICLE SIZE ANALYSIS

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 you should know before the experiment?

- 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?
- How to carry out **gyratory milling**?
- How to determine particle size distribution by **sieving**?

4. Schematic view of experimental procedure

Traditional ceramic powder WET milling in ball mill

↓

Liter weight measurement by picnometer

↓

Drying of the slurry in the oven

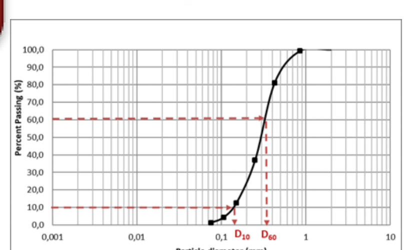
↓

DRY milling in gyrotary mill of preperated traditional slurry

↓

Particle Size analysis via sieving

→



Advanced Ceramic powder wet milling in planetary mill

↓

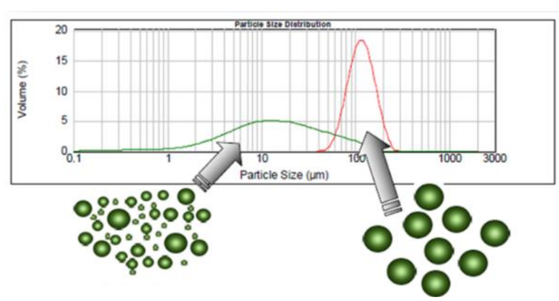
Transferring the slurry to the evaporation flask

↓

Drying of the slurry with rotary evaporator

↓

Particle Size analysis by using Laser Diffraction instrument



5. Equipments and materials

For 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

For Dry milling of ceramics;

Gyratory Mill, Sieve 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, sieving & 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 30%wt	Kaolin 20%wt	Clay 50%wt
Kaolin 25%wt	Clay 10%wt	Kaolin 30%wt
Na-felspar 20%wt	Ukraine Clay 20%wt	Calcite 10%wt
Pegmatite 25%wt	Na-felspar 50%wt	Pegmatite 10%wt

Report Sheet-I

Name & Surname:

Group:

Milling part of traditional ceramic

Materials Composition&Amount (gr)

1. ……………………………………..
2. ……………………………………..
3. ……………………………………..
4. ……………………………………..

Total amount of powder mixture …… (gr)

Defflocculant: ……. (% 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: ………………. (lt/gr)

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: ……………………………

Sieve Analysis

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

QUESTIONS-I

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

2) (a) Using the sieve analysis data provided, plot the particle size distribution curve by graphing the cumulative percentage passing versus the sieve size (on a logarithmic scale for the x-axis). (b) Discuss how sieve analysis and laser diffraction differ in their working principles, and explain what types of materials or particle size ranges are better suited for each method, as well as how the choice of technique may influence the interpretation of material properties.

EXPERIMENT 2

DRY PRESSING AND GLAZING

1. Objective of the experiment

- To demonstrate granulate traditional ceramic powders.
- To demonstrate shaping of wall tiles by uniaxial **pressing.**
- To evaluate the effects of pressing **pressure** and compact **thickness** on green density.
- To apply a **glaze** and explain the key parameters for obtaining decent glazes.

2. What you should know before the experiment?

2.a Dry Pressing

- 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?

2.b Glazing

- What is glaze and 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 and what does deflocculant provide?

3. What will you learn during the experiment?

- 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 and how to control compaction defects?

- Why is frit used in glazes?
- 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

2.a

Granulation of powders

↓

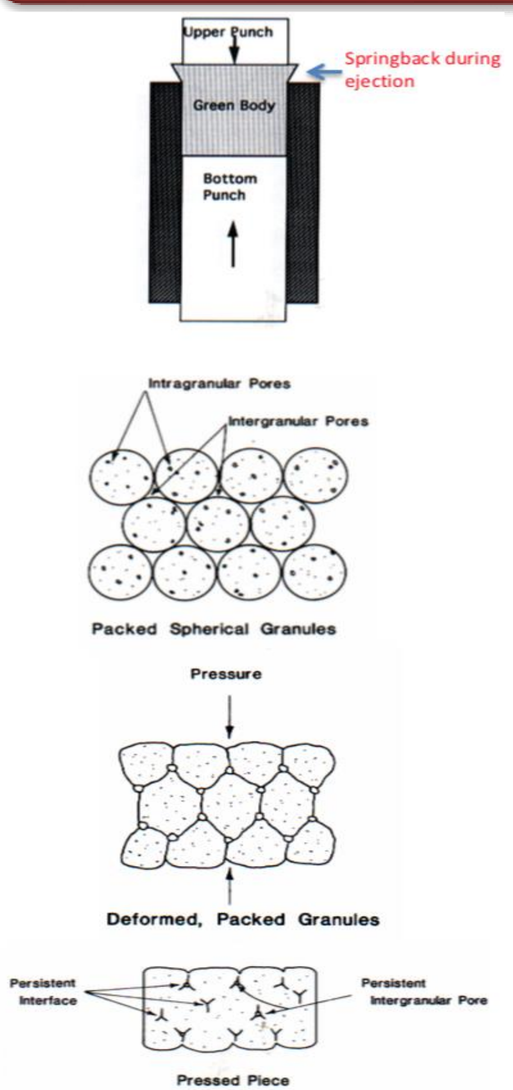
Weighing the powder, filling the die and pressing at the designated pressure

↓

Ejecting the compact from the die

↓

Measure and record the sample's mass and dimensions



2.b

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

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

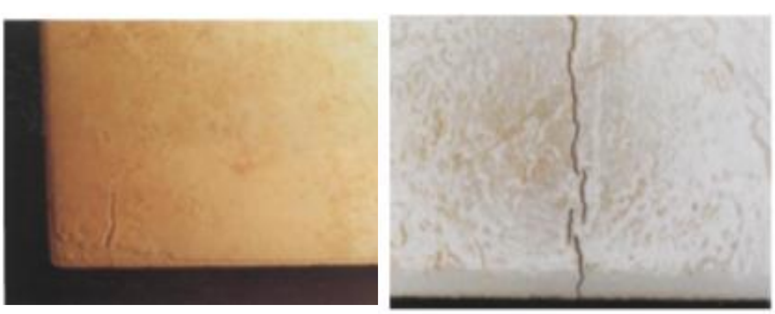
5. Equipment and materials

- Ceramic powder (granulated),
- Pressing dies and mechanical press
- Glaze slurry (commercial)
- Marsh cone apparatus
- Spraying setup and ragle apparatus
- Drying oven; Fast firing furnace

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

- Good powder flow is essential for reproducible volumetric filling, a uniform density of the fill and a rapid pressing rate.
- Hard granules resist deformation and are difficult to change shape, trap residual pore, thus reduce product strength.
- The compact must withstand ejection and handling without fracture and should be free of defects.
- Using lubricant during pressing is important to mitigate friction during compaction and ejection.
- To minimize defect formation, maintain a small holding pressure during ejection process.
- De-air the powder bed prior to compaction to reduce trapped air.
- Criteria for materials' selection
- Milling parameters
- Application techniques
- Firing
- Body-glaze interactions

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



Report Sheet-II

Name & Surname: Group:

2.a Dry Pressing - Measurements

	Pressure (bar)	Radius (cm.)	Thickness (cm.)	Weight (g.)	Green Density (g/cm^3)
Average					
Average					
Average					

!!! Please calculate the green density based on the data obtained above and plot the **pressure-green density graph**.

2.b Glazing

Glaze Application Techniques

1) …………………………………………………

2) …………………………………………………

Glaze Preparation and Application

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)

QUESTIONS-II

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

2) (a) How do the rheological properties, including viscosity, of the glaze affect the application process and the final quality of the glazed product? (b) What additives are used to adjust these properties, and what are their specific purposes?

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 you should 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

↓

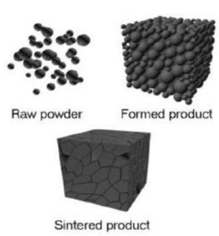
Sintering

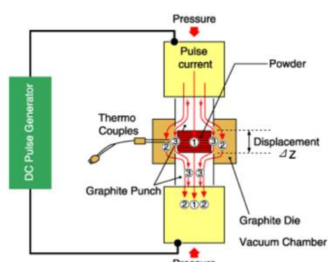
↓

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

⇩

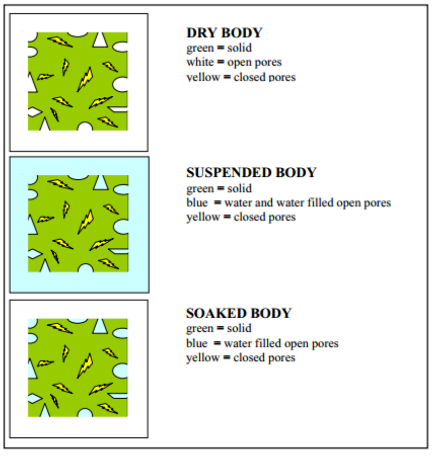
Weight the sample in water (W_w)

⇩

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.

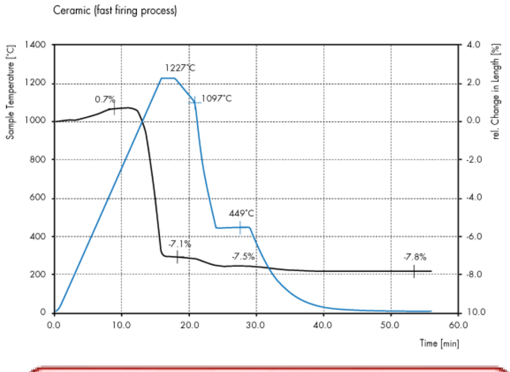
- **For conventional sintering:** the milled and sieved samples prepared in Exp-1 and Exp-2, graphite tools (paper, die, punch, blanket etc) for sintering, spark plasma sintering furnace.

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

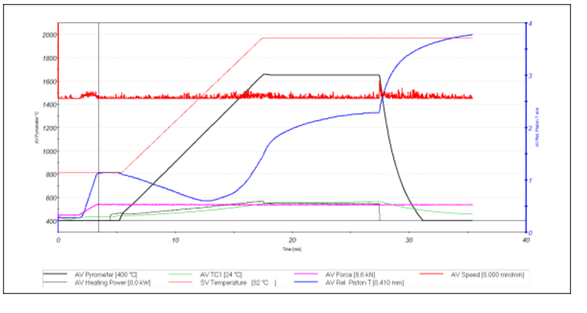
6. Important points/hints for the equipments and/or results obtained from the analyses.

- 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

Report Sheet III

Name & Surname:

Group:

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: ………………

QUESTIONS III

1) 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?

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

1. Objective of the Experiment

For Piezoelectric Part:

- To determine a ceramic sample's **relative dielectric constant (ε_r)** by measuring its capacitance (C) at a specified frequency.
- To correlate the measured ε_r with the material's composition, microstructure (grain size, porosity), and electrode contact/area.

For Thermoelectric Part:

- To demonstrate both the **Seebeck effect** and the **Peltier effect.**
- To calculate the **Seebeck coefficient (S)** from ΔV–ΔT data and interpret its link to material performance.
- To introduce the role of **ceramic thermoelectric materials** in high-temperature and harsh environments, and to highlight their applications in **sensors, cooling, and renewable energy harvesting**.

2. What you should know before the experiment?

For Piezoelectric Part:

- Capacitance and parallel plate model;

$$K = \frac{tC}{\varepsilon_0 A}$$

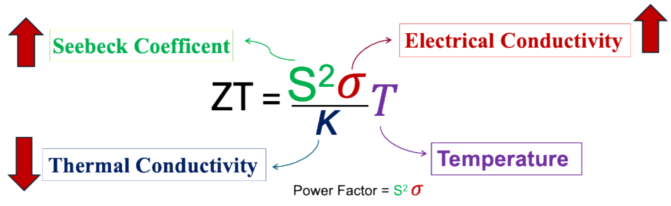
A is the electrode area t is the sample thickness, and ε_0 is the vacum permittivity.

- Relative dielectric constant εr and tangent loss concepts.
- Polarization mechanism
- Dielectric ceramics and sub-gropus
- Piezoelectricity

2. What you should know before the experiment?

For Thermoelectric Part:

- **Seebeck effect (S = −ΔV/ΔT):** A temperature difference across a material generates a voltage.
- **Figure of merit ($ZT = S^2\sigma T/\kappa$):** Thermoelectric performance depends on Seebeck coefficient (S), electrical conductivity (σ), and thermal conductivity (κ).



- **Semiconductors:** Difference between **n-type** (electron carriers) and **p-type** (hole carriers). The sign of the Seebeck coefficient indicates the carrier type.
- **Material requirements:** Good thermoelectric materials need **high S, high σ, and low κ.**
- **Ceramic thermoelectrics:** Oxide ceramics such as ZnO, $CaMnO_3$, $SrTiO_3$, MnO_2, cobaltites are stable at high temperatures and in oxidizing atmospheres.
- **Applications:** Thermocouples, high-temperature sensors, waste-heat recovery, and renewable energy harvesting.

3. What will you learn during the experiment?

For Piezoelectric Part:

- Using the LCR meter (impedance bridge): Do the open/short (and, if available, load) compensation and then zero the instrument before measuring.
- Setting up the parallel-plate fixture: Make good, flat contact between electrodes and sample.
- Measuring across frequency: **Measure capacitance (C).**
- Use these data to calculate ε_r with the parallel-plate formula.
- Understand where piezoelectric and thermoelectric materials fit in the “smart materials”. In this lab, you only measure dielectric properties; piezo/thermo topics are for conceptual context.

3. What will you learn during the experiment?

For Thermoelectric Part:

- Understand the **thermoelectric effect** (Seebeck and Peltier) and its role in energy conversion.
- Calculate the **Seebeck coefficient (S)** from experimental ΔV–ΔT data.
- Recognize how **ceramic processing factors** (e.g., density, porosity, doping) influence thermoelectric properties.
- Identify key **applications** such as thermocouples, cooling devices, and waste-heat recovery systems.
- Discuss the potential of thermoelectric materials in **renewable and sustainable energy technologies**.

4. Background

For Piezoelectric Part:

- Advanced engineering ceramics at high temperatures, serve not only structural roles but also “smart” functions.Constitute a class of materials that, thanks to their low density, high elastic modulus and hardness, chemical/oxidative stability, and microstructural integrity.
- Within this class, thermoelectric and piezoelectric ceramics are two key subgroups that enable direct energy conversion. Thermoelectrics rely on the Seebeck effect, which converts a temperature gradient into an electrical potential.
- Piezoelectric ceramics, by contrast, convert mechanical stress into electric charge (direct effect) and electric field into strain (converse effect).

4. Background

Piezoelectricity – Principles and Concepts

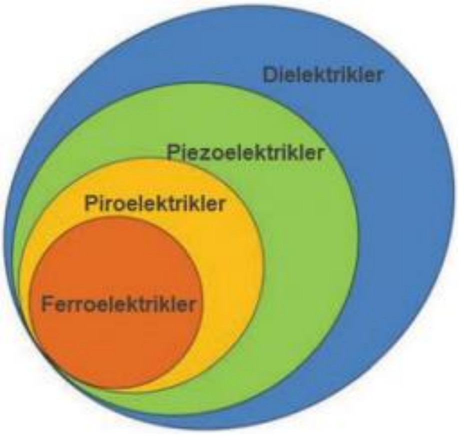


Figure 1. Sub-groups of dielectric materials

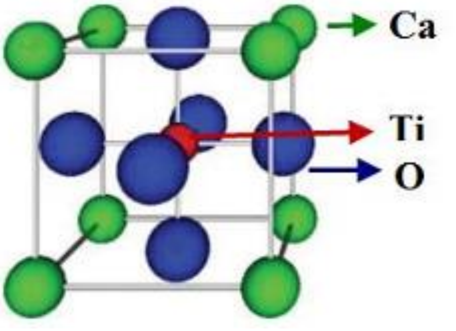
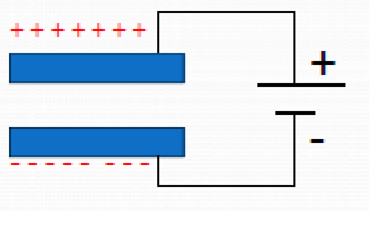
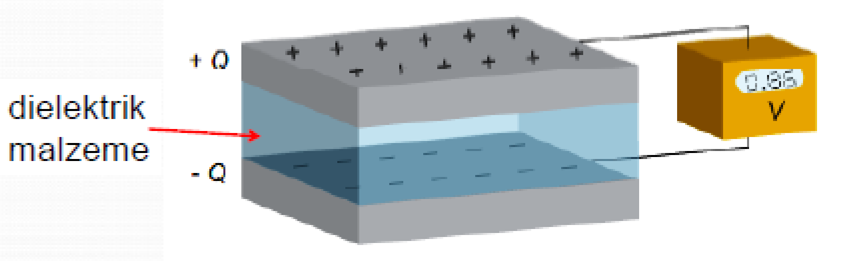


Figure 2. Perovskite crystal structure

Capacitance and Capacitor Concepts



The charge stored on the plates **is retained** after the applied voltage is removed ⇨ It is the ability to store charge

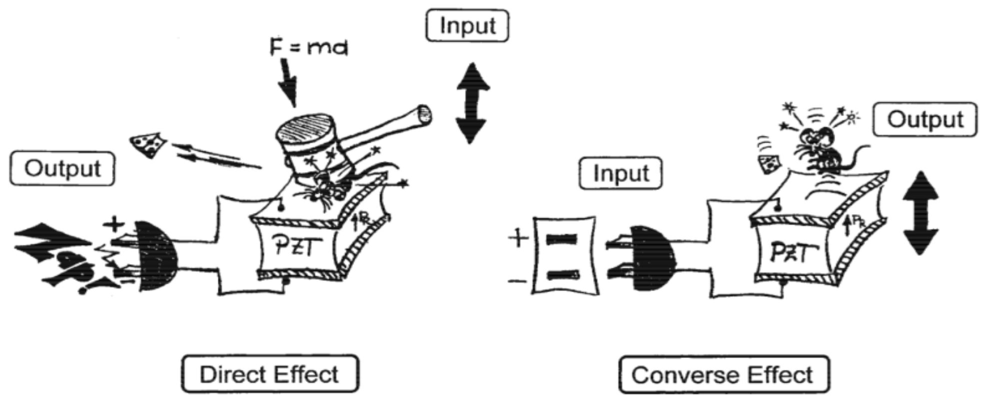


Q = C.V

$$C = \varepsilon_r \varepsilon_o A / L = \varepsilon A / L$$

A: Surface area of the plates (m²)
L: Distance between the plates (m)
ε: Dielectric permittivity of the material between the plates (F/m)
ε_0 :Vacuum permittivity (8.85×10^{-12}F/m)
ε_r :Relative permittivity (dielectric constant) of the material between the plates
Relationship: $\varepsilon_r=\varepsilon/\varepsilon_o$

4. Background



Mechanical Energy ⇨ **Electrical Energy**

Electrical Energy ⇨ **Mechanical Energy**

$P_i = d_{ijk}\,\sigma_{jk}$ (Direct Piezoelectric effect) **$\epsilon_{ij} = d_{kij}\,E_k$ (Inverse Piezoelectric effect)**

•Pi: Polarization generated **along** $i$in response to the applied stress; units **C/m²**.
•d_{ij} :Piezoelectric coefficient relating $P_i$to stress σ_j)or strain $S_i$to field E_j ;(units **C/N** (equivalently **m/V**).
•σ_{jk} :Stress tensor; units **N/m² (Pa)**.
•ε_{ij} :Strain produced (dimensionless, **m/m**) in a given crystal orientation under an electric field applied in the k-direction (when written as $\varepsilon_{ij}(E_k)$
•E_k :Electric field applied in the k-direction; units **V/m**.

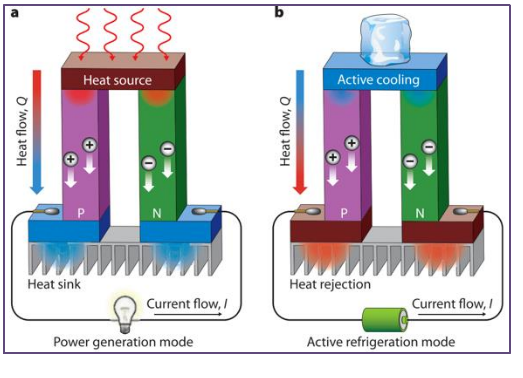
REFERENCES

A.J. Moulson, J.M. Herbert, *Electroceramics: Materials, Properties, Applications*, second ed., John Wiley & Sons, Chichester, 2003

For Thermoelectric Part:

Thermoelectric Effect and Its Importance in Energy Harvesting

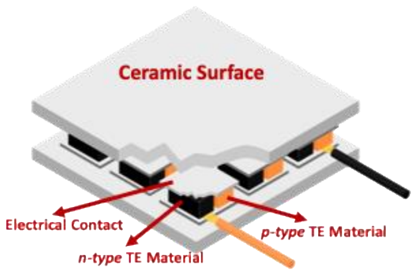
- **Definition:** The thermoelectric effect allows direct conversion between heat and electricity, based on the Seebeck and Peltier phenomena.
- **Energy relevance:** Large amounts of waste heat are released in daily life and in industry (e.g., engines, furnaces, turbines). Thermoelectric devices offer a solid-state, maintenance-free method to **harvest this wasted energy**.



4. Background

Thermoelectric Effect and Seebeck Coefficient

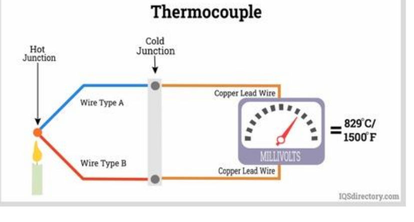
- The **Seebeck coefficient (S)** measures the voltage produced when a temperature difference is applied across a material.
- Along with electrical and thermal conductivity, *S* defines the material's thermoelectric performance (ZT).
- High *S* values are essential for both **efficient energy harvesting** and **sensitive thermal sensing** applications.



Thermoelectric Module

Importance of Thermoelectric Properties in Ceramic Materials

- **Temperature range:** Many thermoelectric ceramics (ZnO, $SrTiO_3$, cobaltites) are stable and functional at **500–1000 °C**, far beyond the safe limit of conventional semiconductors.
- **Industrial sectors:** These ceramics are explored for waste-heat recovery in **steelmaking, glass production, cement plants, and automotive exhaust systems**.
- **Functional ceramics:** Beyond power generation, the Seebeck effect in ceramics supports **temperature sensors, gas sensors, and multifunctional electronic components**.



5. Schematic view of experimental procedure

For Piezoelectric Part:

Prepare the sample. Ensure both faces are clean, flat, and parallel;

↓

Measure the electrode area (A) and the thickness (t) (average at least three points).

↓

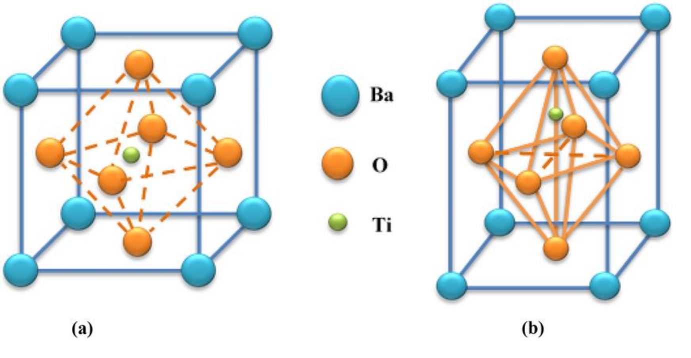
Set up the instrument. Connect the guarded parallel-plate fixture to the LCR meter.

↓

Record capacitance (C) values

↓

Compute εr. Convert all quantities to SI units and calculate the relative dielectric constant using εr



5. Schematic view of experimental procedure

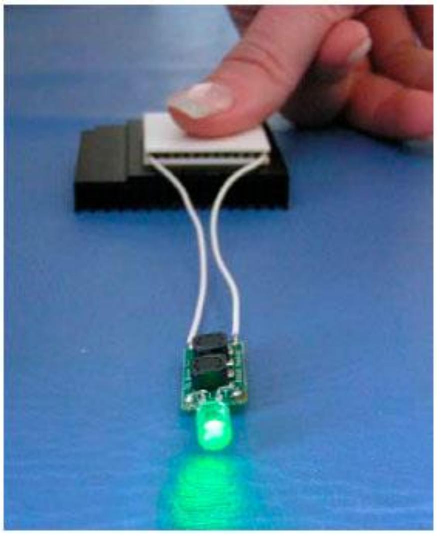
For Thermoelectric Part:

Demonstrate the Peltier effect

Connect the module to a battery. Observe heating on one side and cooling on the other.

Set up for Seebeck measurement

Disconnect the battery. Attach thermocouples to hot and cold sides. Connect module leads to a digital multimeter (open-circuit).



Apply temperature difference

Heat one side (e.g., hot plate) while keeping the other cooled (heat sink/fan). Record T_hot, T_cold, and Voc.

Analyze results

Plot Voc vs ΔT.
Determine the slope → Seebeck coefficient (S = ΔV/ΔT).

6. Equipments and materials

For Piezoelectric Part:

LCR meter, Ag paste, micrometer, samples with different geometries

For Thermoelectric Part:

Thermoelectric (Peltier/TEG) modüle, Digital multimeter, Thermocouples, Heat source (hot plate or heater), Heat sink or fan (for cooling), battery

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

For Piezoelectric Part:

During the experiment and reporting period, consider the relationship among the terms: dielectric constant, tangent loss, electrode area and sample's thickness according to parallel plate model.

For Thermoelectric Part:

Ensure **good thermal and electrical contact**; poor contact leads to errors.

Record **ΔT and Voc together** to correctly calculate Seebeck coefficient ($S = -\Delta V/\Delta T$).

The **sign of S** indicates carrier type: positive → p-type, negative → n-type.

Wait for **steady temperature conditions** before taking measurements.

Report Sheet IV

Name & Surname:

Number:

Group:

Experiment-5 (Piezoelectric)

Quick Fill-ins

Piezoelectricity requires a structure that is…………

Direct piezoelectric effect:
Applied………..→ generated………..

Converse piezoelectric effect:
Applied…..........→ induced…………..

Order the **polarization mechanisms** from fastest to slowest response:
_____ > _____ > _____ > _____.

Measurement Parameters

The experiment was performed at

…….kHz, ……..kHz, ……..kHz.

If the **thickness t is doubled**, the relative dielectric constant **ε_r becomes** …… times its original value.

If the **area A is doubled**, the relative dielectric constant **ε_r becomes** …… times its original value.

Experiment-5 (Thermoelectric)

Quick Fill-ins

Seebeck effect: Applied ……→ generated…….
The reverse of it is the ………… **effect**.

The **sign** of the Seebeck coefficient shows the …….. type: *positive* → ……, *negative* → ……….

The **slope** of the ……....vs ……....graph gives the Seebeck coefficient.

High ……...and ……....but **low** ……....are desired for **good thermoelectric performance**.

Measurement Parameters

Measurements with Battery Connection:

- Battery voltage: _____ **V**
- Measurement time: _____ **min**
- Hot side temperature: _____ **°C**
- Cold side temperature: _____ **°C**

Polarity change: After reversing polarity:

- Measurement time: _____ **min**
- hot side = **…… °C**, cold side = **….. °C**.

Measurements with Open-Circuit Connection:

- Temperature differences applied: _____ **°C, _____ °C, _____ °C**
- Measured open-circuit voltages (Voc): _____ **mV, _____ mV, _____ mV**

Table1. Values Used to Calculate the Dielectric Constant

Sample	Capacitance	Thickness	Average Thickness	Calculated Area	Calculated Dielectric Constant	Frequency (kHz)
1		t1:				1kHz:
		t2:				100kHz:
		t3:				1000kHz:
2		t1:				1kHz:
		t2:				100kHz:
		t3:				1000kHz:
3		t1:				1kHz:
		t2:				100kHz:
		t3:				1000kHz:
4		t1:				1kHz:
		t2:				100kHz:
		t3:				1000kHz:

Graph1. Relationship between Temperature Difference (ΔT) and Generated Voltage (Voc)

Calculated Seebeck coefficient: S = _______ mV/K

QUESTIONS IV

1) Using the parallel-plate model, relate the calculated relative dielectric constant (εr) of your sample to porosity, grain size, electrode contact/guarding, and surface cleanliness. From the literature, compile typical εr ranges (and, if available, loss tangent tanδ only for comparison) for similar ceramics (e.g., Al_2O_3, $BaTiO_3$, PZT, KNN) and justify the expected εr range for your own specimens.

2) (a) Identify the main renewable energy sources currently used worldwide. (b) Estimate their role in the global energy mix (which ones dominate, which are emerging). (c) How could thermoelectric technology be integrated into the renewable energy landscape? Discuss its potential role, limitations, and possible applications in the future.

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 you should 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**.

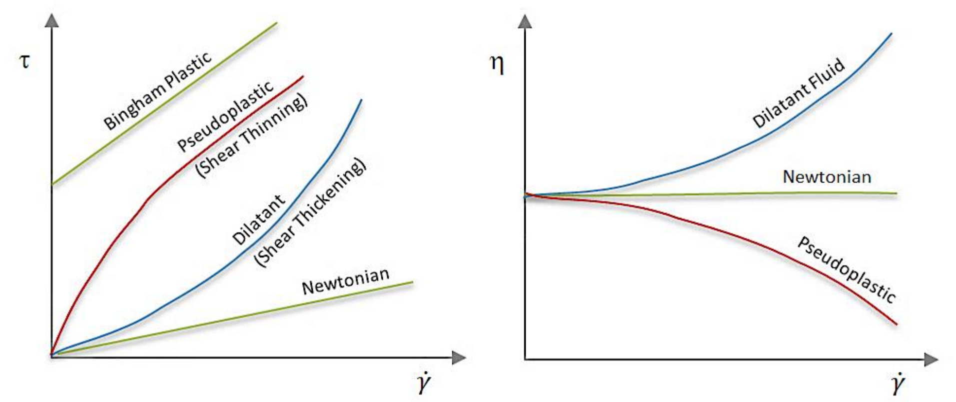


Fig. 1. Schematic a) flow and b) viscosity curves of Newtonian and non-Newtonian materials

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).**

4. Background

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.

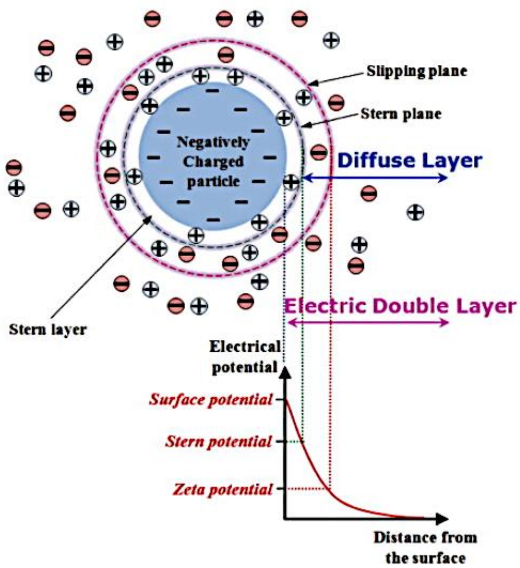


Fig. 2. Diagram of electric double-layer

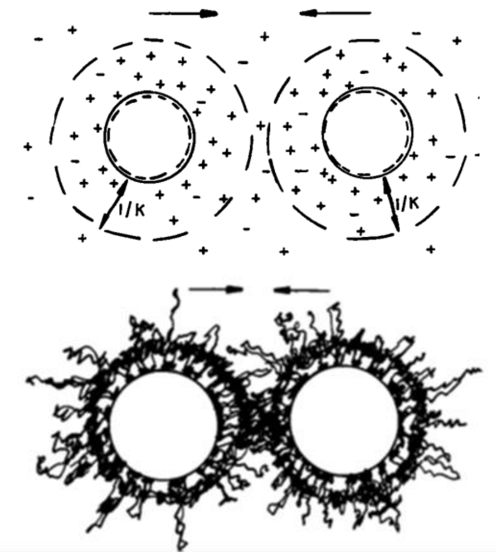


Fig. 3. Schematic representation of electrostatic and steric stabilization

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**.

4. Background

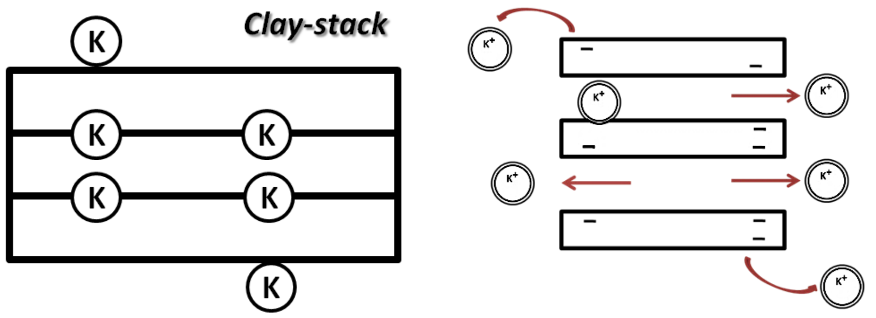


Fig. 4. Kaolin particles a) before and b) after dispersion

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.**

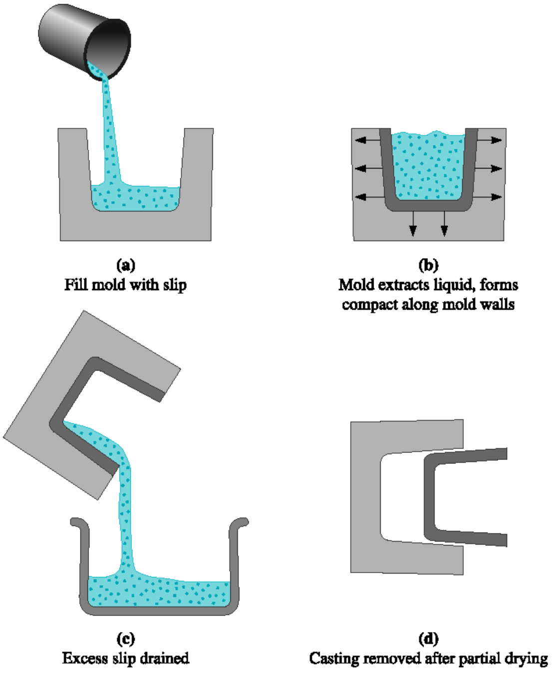


Fig. 5. Slip casting process

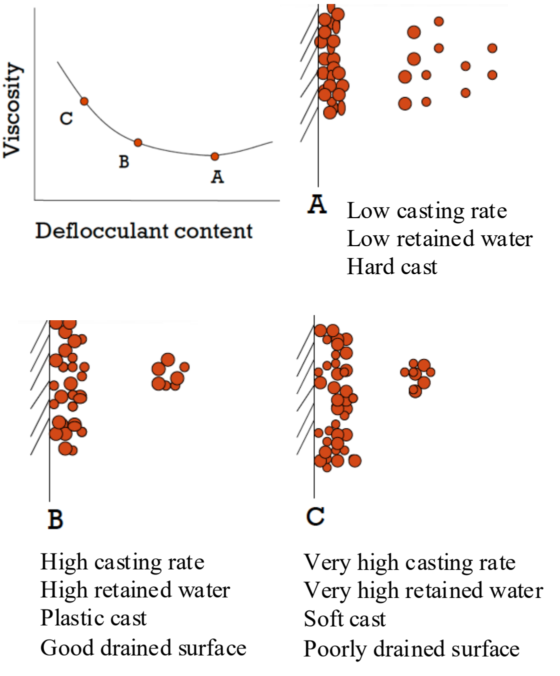


Fig. 6. Effect of slip structure on casting

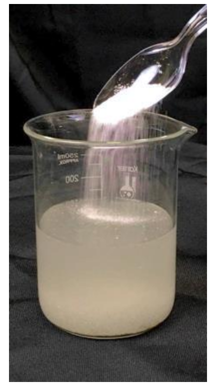
Reed, J.S. (1995). Principles of Ceramic Processing (2nd Ed.) New York: John Wiley & Sons, Inc.
Lee, J.K., Ko, J., and Kim Y.S. (2017). Minerals, 7 (165), 1-11.
Jain S.K. and Shailesh K. Jain. (1986). Conceptual Chemistry Volume-I For Class XII, New Delhi: S. Chand Company Pvt. Ltd.
Napper, D.H. (1983). 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 V

Name & Surname: **Group:**

Slurry preparation

Materials Composition & Amount (g)

1. ……………………………………..

2. ……………………………………..

Solid:liquid mass ratio: ……… (%)

Stirring speed: ……………. (rpm)

3. Deflocculant: ....

(Hint: use Table 1 on 2nd page to note partial and total deflocculant amounts)

Total deflocculant amount …….…... (g)

Total deflocculant content (solid:solid mass ratio) …….…. (wt%)

Adjusting viscosity by deflocculant content

Time to read accurate cP value: …….. (s)

Viscosity of the rested slurry: ………. (cP)

Interval stirring duration: …….. (min)

Viscosity after stirring: ……….. (cP)

Final viscosity …….…… (cP)

(Hint: use Table 2 and 3 to note defloc. contents and viscosity values)

Slip casting

Type of molds: ……………………..

Drying periods: ………………… (min)

Viscosity (cP) - Cum. defloc. content (wt%)
(Hint: use the data on Table 3)

Avg. wall thickness (mm) - Drying period (min)
(Hint: use the data on Table 4)

Slurry No	Final Viscosity (cP)	Avg. Wall Thickness of Cast at 10th min (mm)
1		
2		

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)

QUESTIONS-V

1. (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?

2. 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?