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The university motto of National Chung Cheng University

積極創新 修德澤人

Strive for Innovation, Cultivate Virtue to Benefit Others

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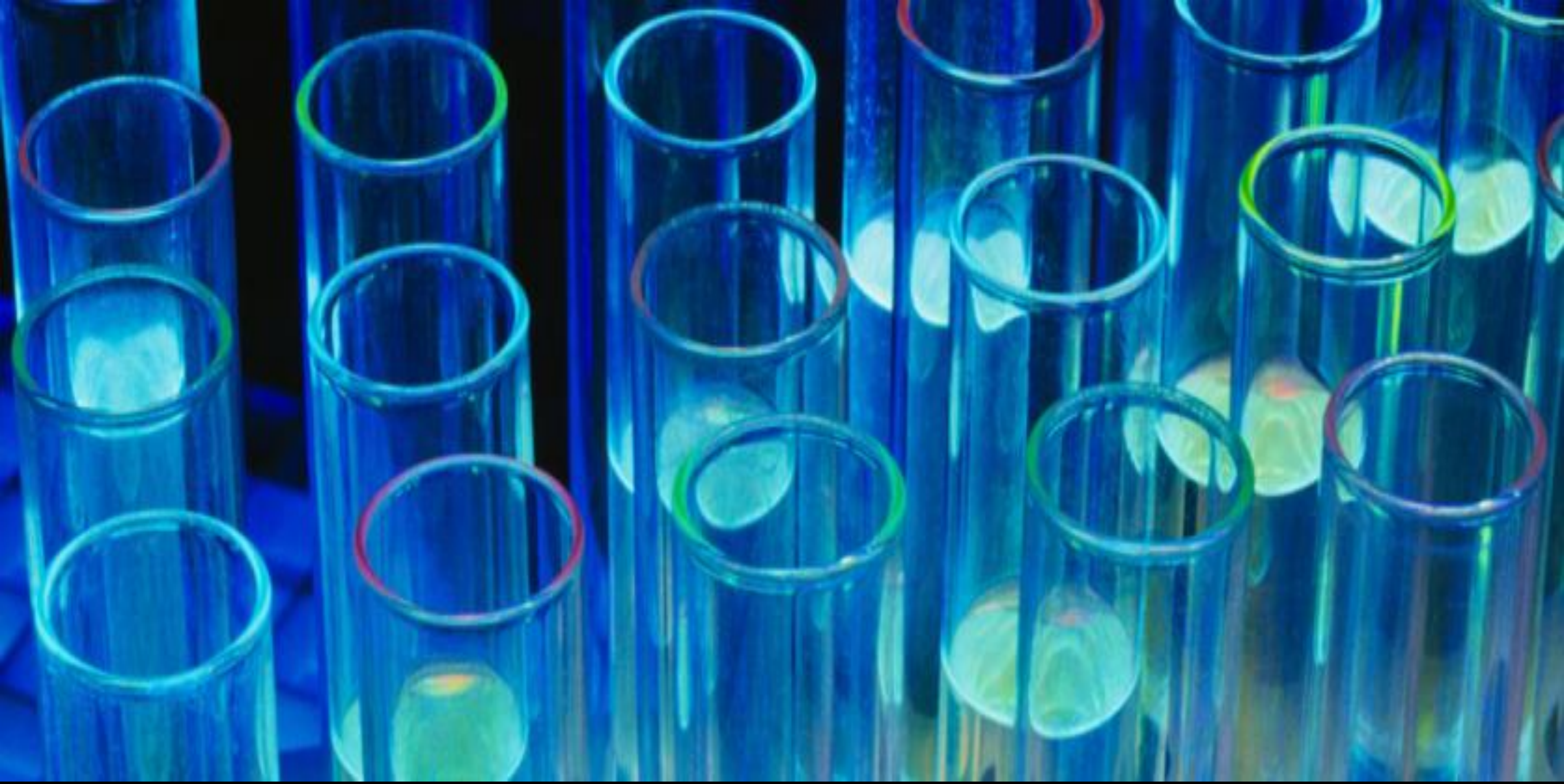

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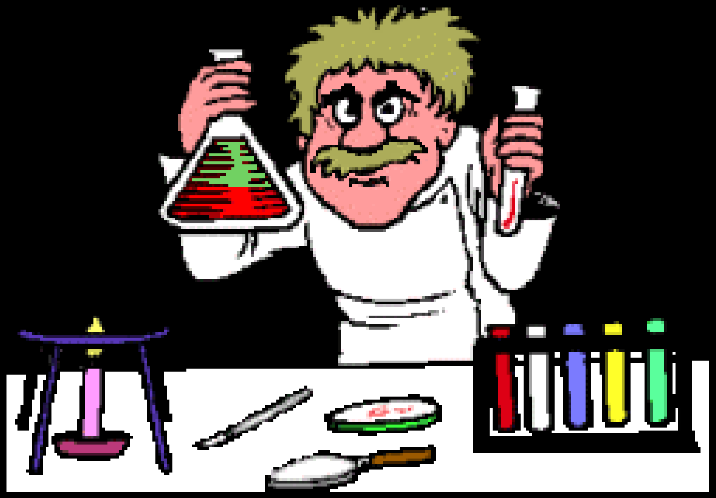
Expt 3 - Microwave-Assisted Extraction of Iron from Sesame Powder: A Green and Energy-Efficient Approach

May 29, 2026 (time required: 2 hours)

1. Objectives

Iron are extracted from sesame powder using conventional heating and microwave-assisted heating, and the extraction efficiencies of the two methods are compared.



2. Principle (1/6)

- **Iron are essential nutrients for both plants and animals and are a key component of hemoglobin. Iron ion deficiency can lead to malnutrition and anemia. The human requirement for iron is highest during periods of growth or blood loss, such as early childhood and pregnancy.**

2. Principle (2/6)

- **Iron must be replaced through daily dietary intake, making their measurement in foods important. However, because iron levels in most foods are close to the detection limit, their quantitative analysis can be relatively complex and challenging.**

6 mg /100 g

2.7 mg /100 g

80 mg /100 g

Figure 1. Common iron-containing foods; from left to right: oats, spinach, and wood ear mushroom.

2. Principle (3/6)

- Metal ions are typically extracted using acidic solutions or chelating agents prior to analysis. In this experiment, iron is extracted from food using a conventional method (e.g., a heating plate) and compared with microwave-assisted extraction. The extracted Fe^{3+} are then reacted with thiocyanate (SCN^{-}) to form an $Fe(SCN)_3$ complex, whose absorbance at 450 nm is measured by UV–Vis spectrophotometry for quantitative analysis.

$$Fe^{3+}_{(aq)} + 3\ SCN^{-}_{(aq)} \longrightarrow Fe(SCN)_{3(aq)}$$

2. Principle (4/6)

Conventional Heating and Microwave Heating

In conventional heating, heat is transferred from an external source to the solution by conduction through the container, and then distributed to the reactants by convection. During this process, part of the energy may be lost to the container or the surroundings.

In contrast, microwave heating directly interacts with polar molecules in the reactants and solvent. Microwave irradiation induces rapid molecular motion and ionic motion, generating heat at approximately 2.45 billion oscillations per second. As a result, microwave heating offers faster heating rates, higher energy efficiency, and more uniform heating than conventional methods.

1 MICROWAVE HEATING PRINCIPLE

Microwaves heat the sample from inside out!

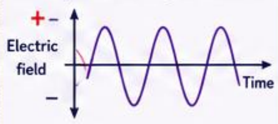
Microwave
2.45 GHz
(2.45×10^9 cycles per second)

WHAT IS A MICROWAVE?

Microwaves are electromagnetic waves with a frequency of

2.45 GHz

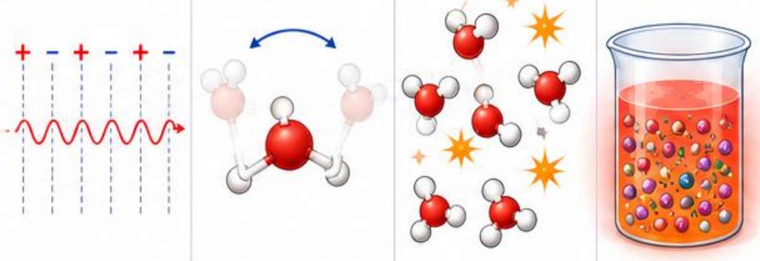
(2.45×10^9 cycles per second)



One cycle ≈ 4.1×10^{-10} s
(0.41 nanoseconds)

HOW MICROWAVES HEAT MATTER

1. Microwaves create an alternating electric field.
2. Polar molecules (especially water) rapidly rotate (dipolar rotation) to align with the field.
3. Molecular collisions and friction occur.
4. The lost energy is converted to heat throughout the entire sample.



FAST • UNIFORM • EFFICIENT

KEY FEATURES OF MICROWAVE HEATING

- ✓ Heat is generated throughout the solution (not just from the surface).
- ✓ Very fast and efficient.
- ✓ More uniform temperature.
- ✓ Ideal for polar solvents (water) and ionic species (Fe^{2+}, etc.).

WHY IT WORKS FOR THIS EXPERIMENT

Microwave heating rapidly increases temperature inside seed cells and solvent, breaking cell walls and enhancing diffusion of Fe^{2+} into solution, leading to higher extraction efficiency.

2 COMPARISON: TRADITIONAL HOT PLATE HEATING vs MICROWAVE HEATING

ITEM	TRADITIONAL HOT PLATE HEATING	MICROWAVE HEATING
Heat Transfer	Heat transferred from bottom to top by conduction and natural convection.	Heat generated throughout the solution by molecular interactions.
Heating Rate	Slow heating (10–30 min to reach extraction temperature)	Very fast heating (2–5 min to reach extraction temperature)
Temperature Distribution	Uneven temperature. Hotter at the bottom, cooler at the top.	Uniform temperature throughout the entire solution.
Energy Efficiency	Lower efficiency (energy lost to environment)	Higher efficiency (minimal energy loss)
Extraction Performance	Slower cell rupture and Fe^{2+} diffusion → Lower extraction	Rapid cell rupture and Fe^{2+} diffusion → Higher extraction
Time & Energy Use	Longer time Higher energy use	Shorter time Lower energy use

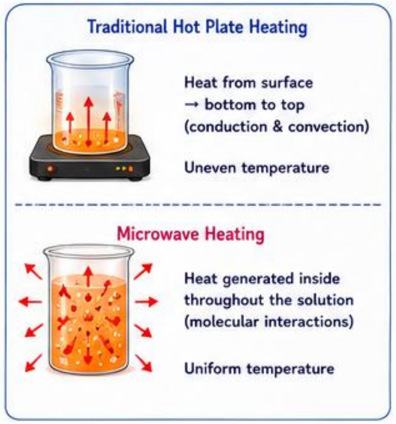
WHY MICROWAVE HEATING IS AN ENERGY-SAVING METHOD?

Saves Energy
Up to 50–80% less energy consumption than hot plate.

Saves Time
Extraction in minutes, not tens of minutes.

Better Performance
Higher Fe^{2+} recovery and reproducibility.

Greener Chemistry
Lower energy use, lower carbon footprint.



Using microwave-assisted extraction with nitric acid (10.0% HNO_3) helps us extract Fe^{2+} from sesame powder more efficiently, faster, and with less energy.

⚠ In this experiment, **10.0% nitric acid (HNO_3)** is used as the extraction solvent. It provides an acidic environment that dissolves iron and helps release Fe^{2+} from the sample.

2. Principle (5/6)

Principles of Microwave Heating

The principle of microwave heating relies on the presence of an electric field. Heat is generated through interactions with molecules via the following two mechanisms:

Dipolar rotation

Polar molecules continuously reorient themselves back and forth as their dipoles attempt to align with the alternating electric field. Friction between rotating molecules generates heat.

2. Principle (6/6)

Principles of Microwave Heating (continued)

Ionic conduction

Free ions migrate through the medium in response to the alternating electric field. Similar to dipolar rotation, collisions and friction among moving species produce heat. As the temperature of the reaction mixture increases, energy transfer becomes more efficient.

3. Equipment and Chemicals (1/2)

Equipment

In cabinet	Provided by TA
Beakers 100 mL × 2	Dropper × 7
Volumetric flask 100 mL × 1	Filter paper × 2
Volumetric flask 10 mL × 3	Cleaning wipes
Thermometer × 1	Plastic wrap
Hot plate × 1	Cuvette × 1
	Microwave oven (1500 W) × 1
	Spectrophotometer × 1

3. Equipment and Chemicals (2/2)

Chemicals

10.0% HNO_3 **	Black sesame powder *
$FeCl_3 \cdot 6H_2O$ **	1.0% KSCN *

* : Corrosive * : Toxic * : Flammable

Ref. : https://pubchem.ncbi.nlm.nih.gov/

4. Experimental Procedures (1/9)

(I) Calibration curve for iron standard solutions (one dataset shared by two groups)

1. **Preparation of Intermediate Standard Solutions**
   - **Stock Solution:** Prepare approximately 40.0 mL of 0.325 mg/mL iron ion standard solution.
   - **Serial Dilution:** Use 1.0% potassium thiocyanate solution to serially dilute the stock solution.
   - **Intermediate Standards:** Prepare 10.00 mL of 0.225, 0.250, 0.275, and 0.300 mg/mL iron ion standard solutions.

4. Experimental Procedures (2/9)

2. Preparation of Working Standard Solutions

- **Further Dilution:** Further dilute the above intermediate standard solutions with **1.0% potassium thiocyanate solution**.
- **Final Volume:** Dilute each solution to 10.0 mL.
- **Working Standards:** Prepare working standards of 0.0225, 0.0250, 0.0275, 0.0300, and 0.0325 mg/mL.

4. Experimental Procedures (3/9)

3. Absorbance Measurement and Calibration Curve Construction

- **Blank Solution:** Use 1.0% potassium thiocyanate solution as the blank.
- **Sample Measurement:** Measure the absorbance of the five working standard solutions: 0.0225, 0.0250, 0.0275, 0.0300, and 0.0325 mg/mL.
- **Wavelength Setting:** Perform the measurement at **450 nm** using a spectrophotometer.
- **Calibration Curve:** Plot the absorbance values against concentration to construct the calibration curve.

4. Experimental Procedures (4/9)

(II) Extraction of iron from sesame powder using a hot plate heating method

1. Sample Preparation
   - **Weighing:** Weigh approximately 5.000 g of sesame powder.
   - **Sample Addition:** Transfer the sesame powder into a beaker containing a stir bar.
   - **Acid Addition:** Add 25.0 mL of 10.0% nitric acid to the beaker.
   - **Mixing:** Stir the mixture to ensure uniform contact between the sesame powder and the acid solution.

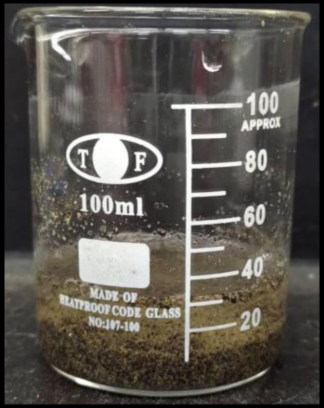


Figure 2. Picture of the solution before heating.

4. Experimental Procedures (5/9)

2. **Hot Plate Heating**
   - **Heating:** Heat the mixture on a hot plate while maintaining the temperature at **40 °C**.
   - **Extraction Time:** Maintain the temperature for 30 minutes to extract iron ions from the sesame powder.
3. **Filtration and Collection**
   - **Filtration:** After heating, perform gravity filtration to remove solid residues.
   - **Extract Collection:** Collect the filtrate as the iron-containing extract solution.
   - **Final Appearance:** An orange-red extract solution should be obtained.

Figure 3. Picture of the filtered solution after heating.

4. Experimental Procedures (6/9)

(III) Extraction of iron ions from sesame powder using a microwave heating method

1. Sample Preparation
   - **Weighing:** Weigh approximately 5.000 g of sesame powder.
   - **Sample Addition:** Transfer the sesame powder into a beaker.
   - **Acid Addition:** Add 25.0 mL of 10.0% nitric acid to the beaker.
   - **Mixing:** Stir the mixture to ensure uniform contact between the sesame powder and the acid solution.

4. Experimental Procedures (7/9)

2. **Microwave Heating Setup**
   - **Plastic Wrap Covering:** Cover the beaker with plastic wrap and **puncture several small vent holes**.
   - **Microwave Heating:** Heat the mixture in a microwave oven at 750 W for 25 seconds.

3. **Filtration and Collection**
   - **Filtration:** After heating, perform gravity filtration to remove solid residues.
   - **Extract Collection:** Collect the filtrate as the iron-containing extract solution.
   - **Final Appearance:** An orange-red extract solution is obtained.

Figure 4. Picture of microwave heating.

4. Experimental Procedures (8/9)

(IV) Quantification of iron ions in sesame powder using the hot-plate heating method

1. Sample Dilution
   - **Sample Pipetting:** Pipette 1.00 mL of the hot-plate-prepared orange-red solution into a 10 mL volumetric flask.
   - **Dilution:** Add 1.0% potassium thiocyanate (KSCN) solution and dilute to the calibration mark.
2. Blank Preparation and Measurement
   - **Blank Solution:** Use 1.0% potassium thiocyanate solution as the blank.
   - **Dilution:** Add 1.0% potassium thiocyanate (KSCN) solution to the flask, dilute the solution to the calibration mark.

4. Experimental Procedures (9/9)

(V) Quantification of iron ions in sesame powder using the microwave heating method

1. Sample Dilution
   - **Sample Pipetting:** Pipette 1.00 mL of the microwave-prepared orange-red solution into a 10 mL volumetric flask.
   - **Dilution:** Add 1.0% potassium thiocyanate (KSCN) solution to the flask, dilute the solution to the calibration mark.
2. Blank Preparation and Measurement
   - **Blank Solution:** Use 1.0% potassium thiocyanate solution as the blank.
   - **Dilution:** Add 1.0% potassium thiocyanate (KSCN) solution to the flask, dilute the solution to the calibration mark.

Microwave Oven Operation Guide

Select Time

Select Power Level (P5)

Stop (Red) / Start (Green)

The microwave oven consists of an upper control panel and a lower door. The control panel includes three main functions: power selection, time setting, and start/stop control.

To adjust the power, press “Power Level” and set it to P5 (750 W).

Microwave Oven Operation Guide

After selecting the required power level, place the sample in a container and cover it with plastic wrap and puncture several vent holes, then put it into the microwave oven.

Set the heating time by entering the desired time on the numeric keypad, or press +10 s to adjust the heating duration.

Microwave Oven Operation Guide

After setting the heating time, press "Start" to begin heating.

If heating needs to be stopped during the process, press "Stop."

After heating is completed, do not remove the container immediately after heating.

Use heat-resistant gloves and wait until the container has cooled down before handling.

5. Precautions (1/2)

1. **Safety:** Standard PPE (lab coat, goggles, and gloves) is mandatory at all times.
2. **Chemical Handling:** Handle all chemicals with care, as they may be toxic or hazardous. Follow all laboratory safety regulations and the instructions of the TAs at all times.
3. **Prohibited Actions:** Removal of any chemicals or laboratory equipment from the lab is strictly prohibited.
4. **Instrument Preheating:** The spectrophotometer must be preheated for 20 minutes before use.
5. **Cuvette Cleaning:** When cleaning the cuvette, clean only the inner surfaces; do not touch the outer surface.

5. Precautions (2/2)

6. **Optical Surface Care:** When handling the cuvette, avoid touching the optical (clear) surfaces, and wipe them with lens tissue before measurement.
7. **Cuvette Alignment:** During measurement, ensure that the orientation marks on the cuvette and the instrument are aligned.
8. **Microwave Safety:** When operating the microwave oven, do not open the door during heating. Wear heat-resistant gloves when handling heated samples.
9. **Odor Control:** Odors may be generated during heating. After heating, place the sample in a fume hood and allow it to cool completely.
10. **Reagent Preparation:** Each two groups require about 120.0 mL of 1% potassium thiocyanate solution. First measure approximately 120.0 mL, and take more if necessary.

6. Experimental data (1/2)

1. **Analytical Wavelength λ : _____ nm**
2. **Standard Fe(III) Solution Concentration : ______ mg / mL**
3. **Plot absorbance (y-axis) versus Fe(III) concentration (x-axis).**

Fe(III) concentration (mg/mL)	Absorbance (A)
0.0325	
0.0300	
0.0275	
0.0250	
0.0225	

6. Experimental data (2/2)

4. Microwave Oven and Hot Plate Stirrer Heating Record Table
(The power of the hot plate stirrer is based on the value indicated on the bottom label of each group's instrument.)

	Microwave oven	Hot plate
Sesame powder mass (g)		
Absorbance of iron ions in sesame powder (A)		
Iron ion concentration in sesame powder (mg/mL)		
Power (W)		
Heating time (s)		
Energy consumption (kJ)		

7. Questions (1/4)

1. **If the cuvette used in this experiment has a path length of 1.0 cm, and 2.0 mL of a 0.4 mg/mL standard iron(III) ion solution is diluted to 10.0 mL according to the experimental procedure, the measured absorbance is 0.686.**
   If the cuvette is replaced with one having a path length of 3.0 cm, and the volume of the standard iron(III) ion solution used is changed to 0.50 mL to prepare the same final solution, what will be the absorbance of the resulting sample?

7. Questions (2/4)

2. **What is the recommended daily iron intake for a normal adult? List three iron-rich foods and indicate the data source.**

7. Questions (3/4)

3. **Based on the experimental data in Table 4, use the iron ion concentration in sesame powder obtained by microwave heating and hot-plate stirring heating to calculate the iron ion content in 100 g of sesame powder, respectively. Compare the results with the labeled iron content of commercially available sesame powder. Calculate the extraction efficiency of the two methods and discuss possible sources of error.**

7. Questions (4/4)

4. **What is the role of iron ions in the human body? What effects occur if the level is too low or too high?**

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