CREST
Center for Renewable Energy and Storage Technologies at KAUST

women & energy

مدينة الملك عبد الله للطاقة الذرية والمتجددة
K•A•CARE

Electrolysis

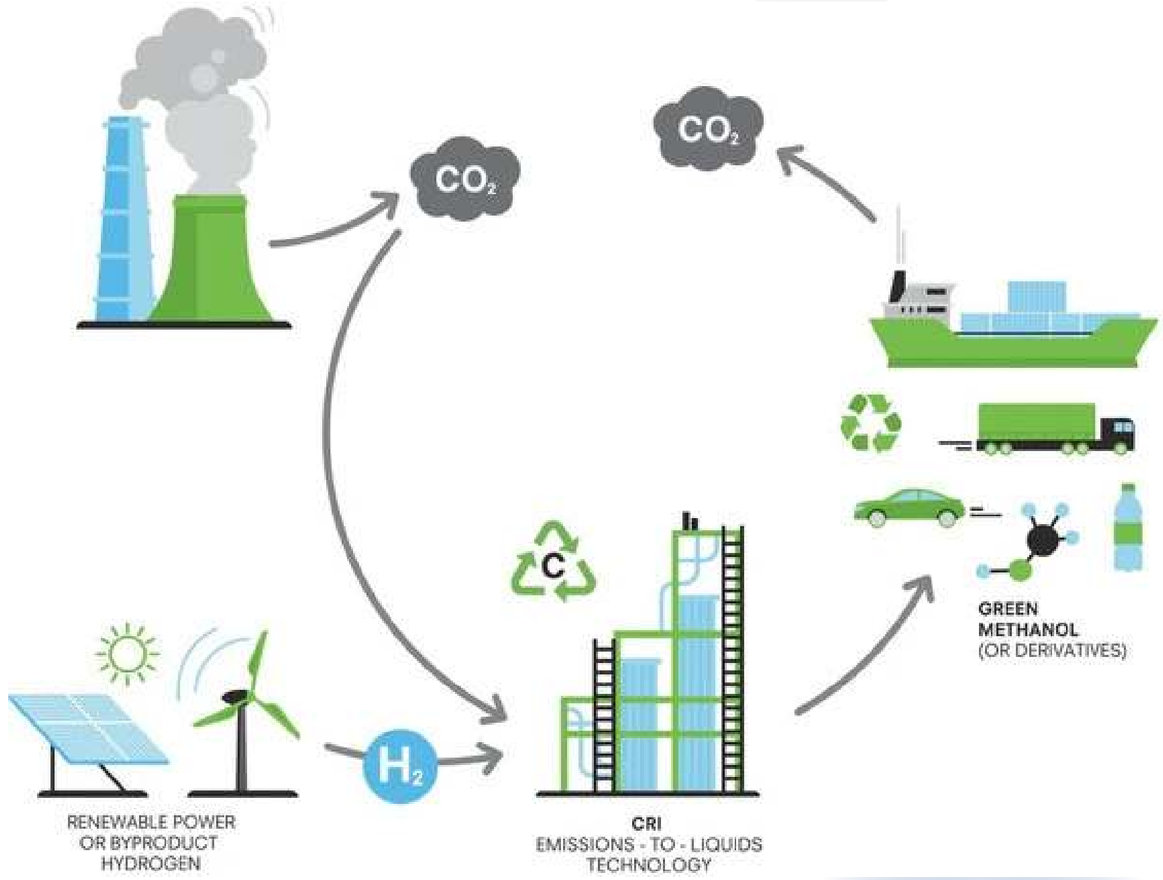
Under The Supervision Of Prof. Huabin Zhang
Under The Mentorship Ph.D Student Abdulrahman Shahad al Gamdi

أكاديمية كاوست
KAUST ACADEMY

جامعة الملك عبدالله للعلوم والتقنية
King Abdullah University of Science and Technology

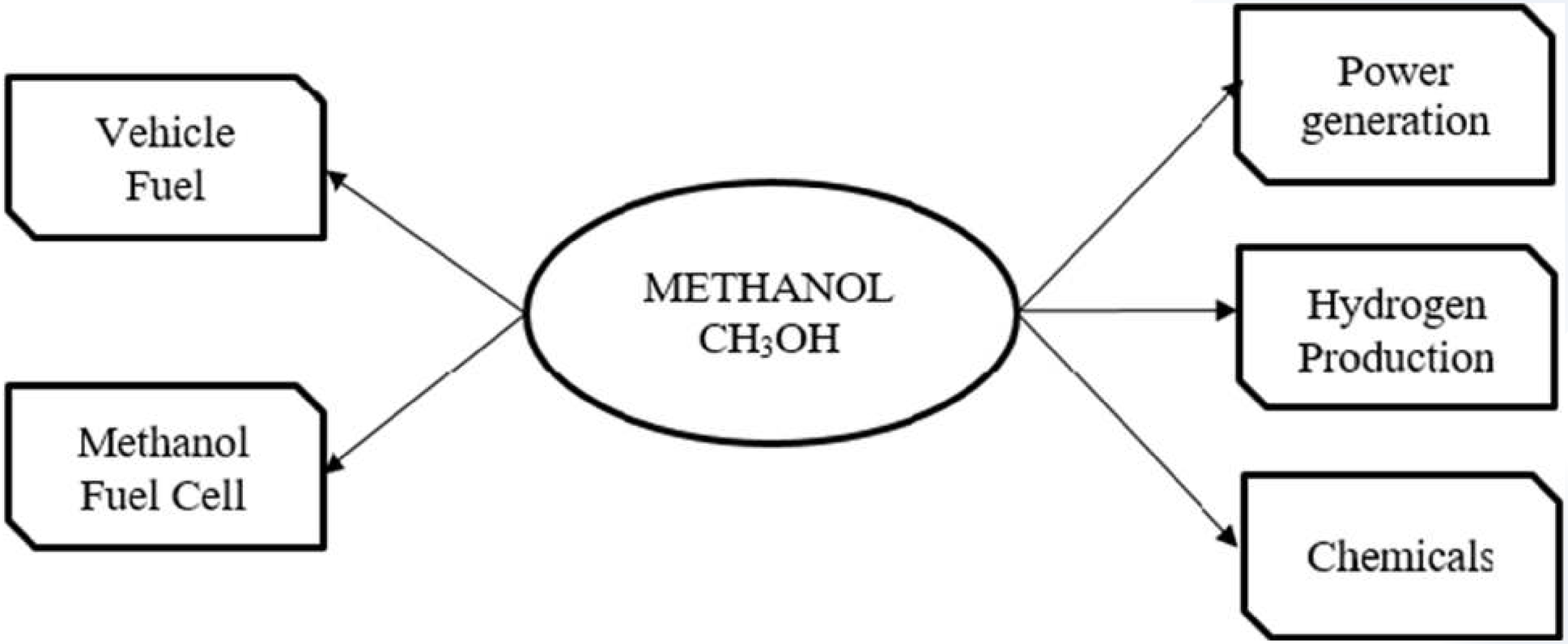
CO_2 Reduction

- Reduces CO_2 emissions by transforming carbon waste into valuable fuels and chemicals.
- Promotes a circular economy through sustainable reutilization of CO_2 as a raw material.
- Enables low emission production when integrated with renewable energy technologies.



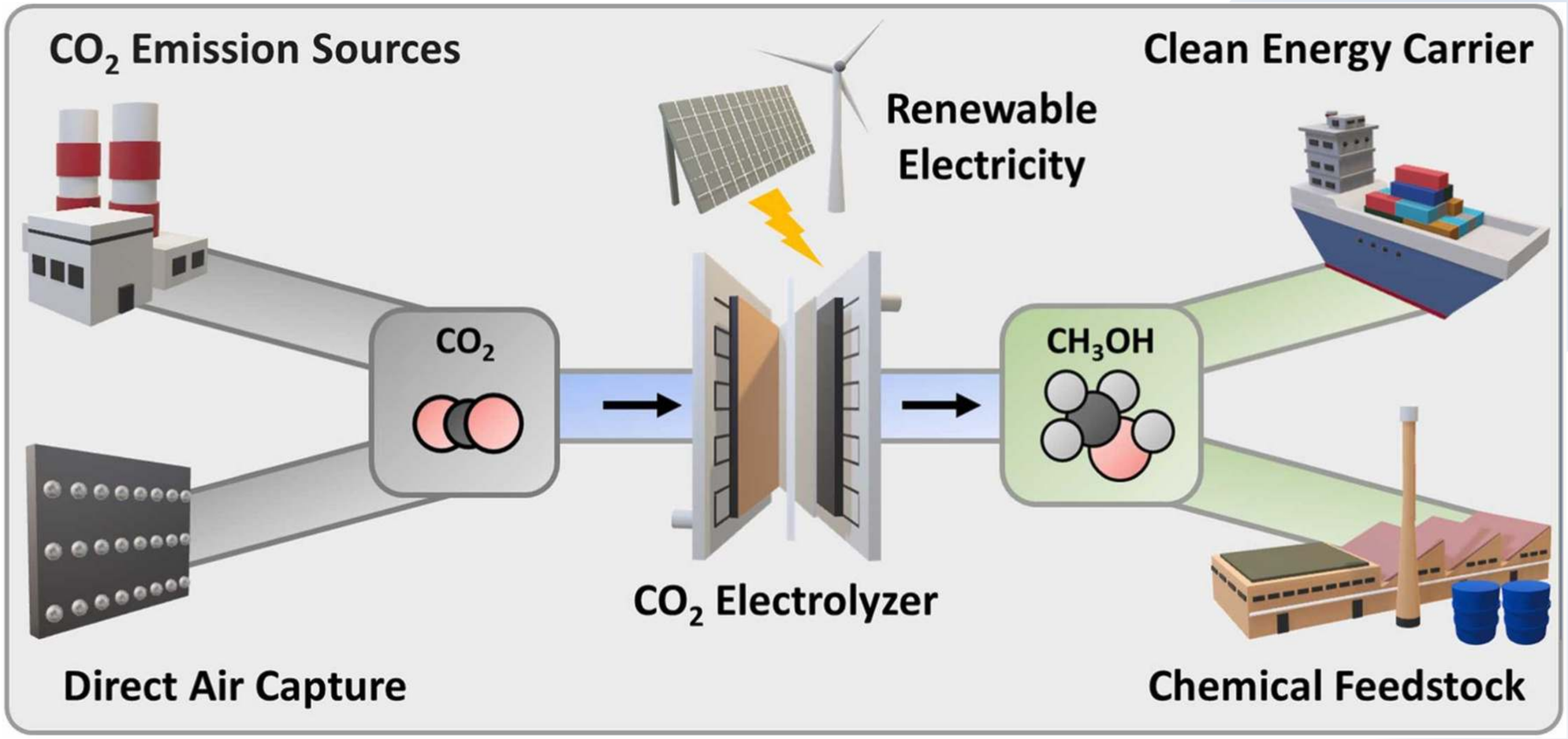
Topics in Catalysis 61.7 (2018): 530–541.

The main uses of methanol as an energy source :



Topics in Catalysis 61.7 (2018): 530–541.

Electrochemical CO_2 Reduction to e-MeOH



Nano Energy, 130, 110099.

Lecture review

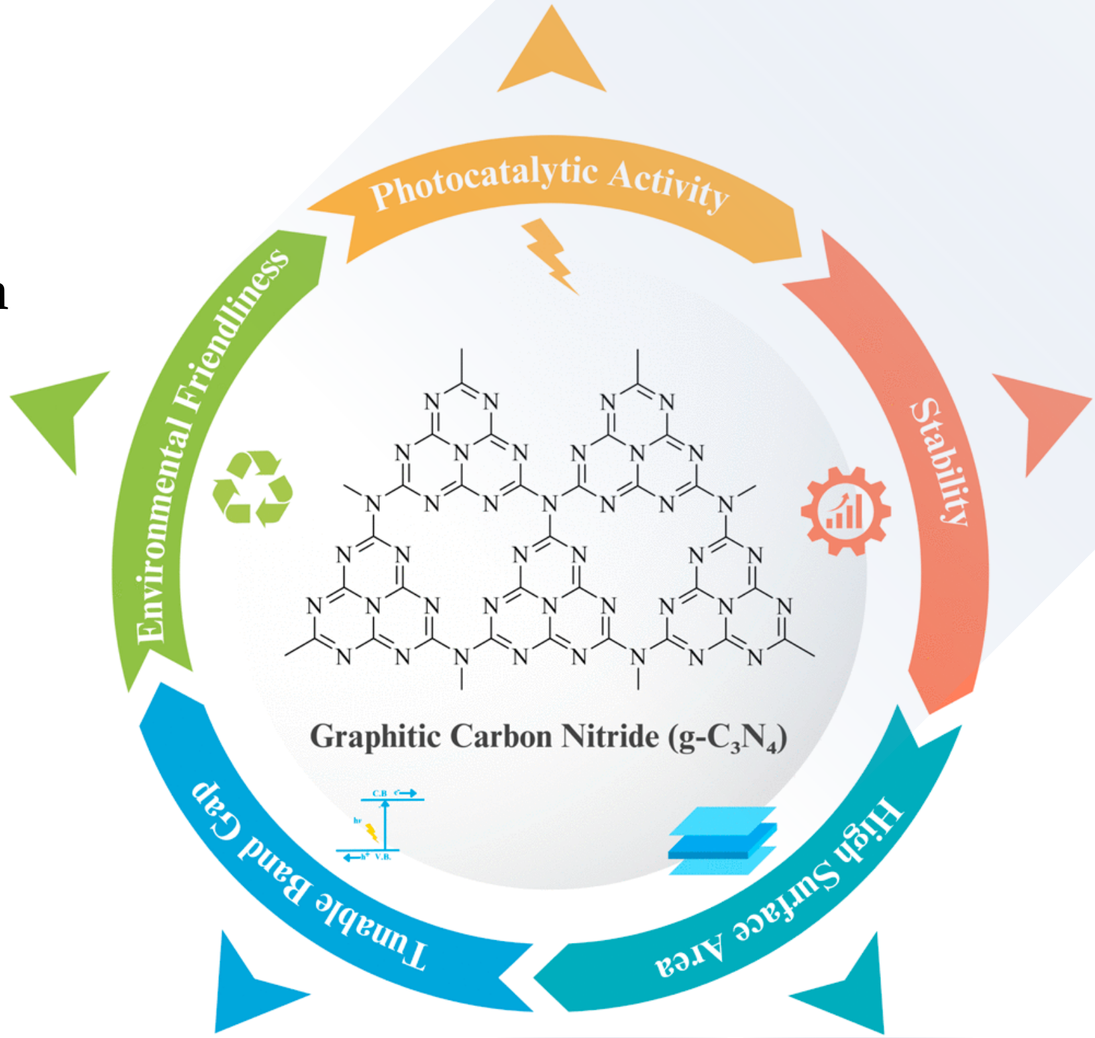
Graphitic carbon nitride ($g-C_3N_4$) is a metal free, polymeric semiconductor composed of carbon and nitrogen.
It has a layered structure resembling graphite.

The unique combination of properties of ($g-C_3N_4$) makes it an ideal candidate for sustainable applications. These properties include:

- Excellent stability
- Ease of synthesis
- Suitable bandgap

Thanks to these features, $g-C_3N_4$ is widely applicable in areas such as:

- Water purification
- Renewable energy production
- Air quality improvement
- Carbon management



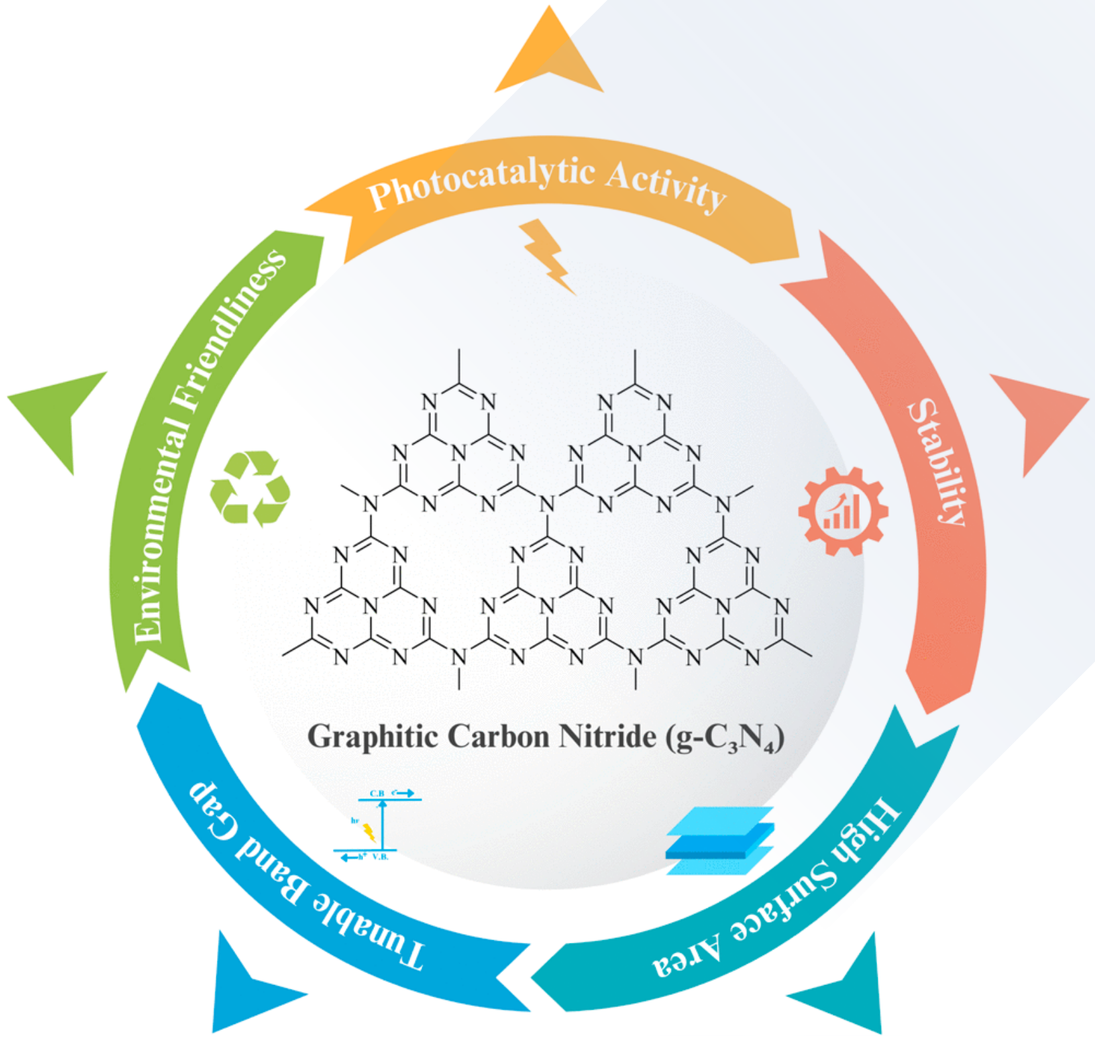
RSC sustainability, 2(2), 265–287.

Lecture review

Role in electrocatalysis

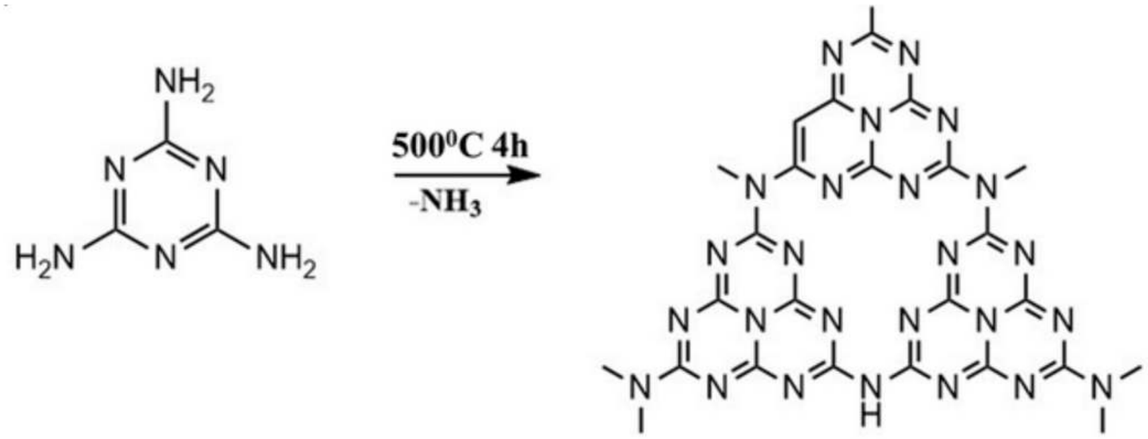
Commonly used as a support material for metal based catalysts (Cu, Ni, Co), Offers several advantages:

- Nitrogen rich surface improves interaction with reactants.
- Partial conductivity facilitates electron transfer.
- Strong interaction with metal enhances:
  - Catalyst dispersion
  - Electron mobility
  - Overall reaction performance



RSC sustainability, 2(2), 265–287.

Synthesis of Graphitic Carbon Nitride ($g-C_3N_4$):



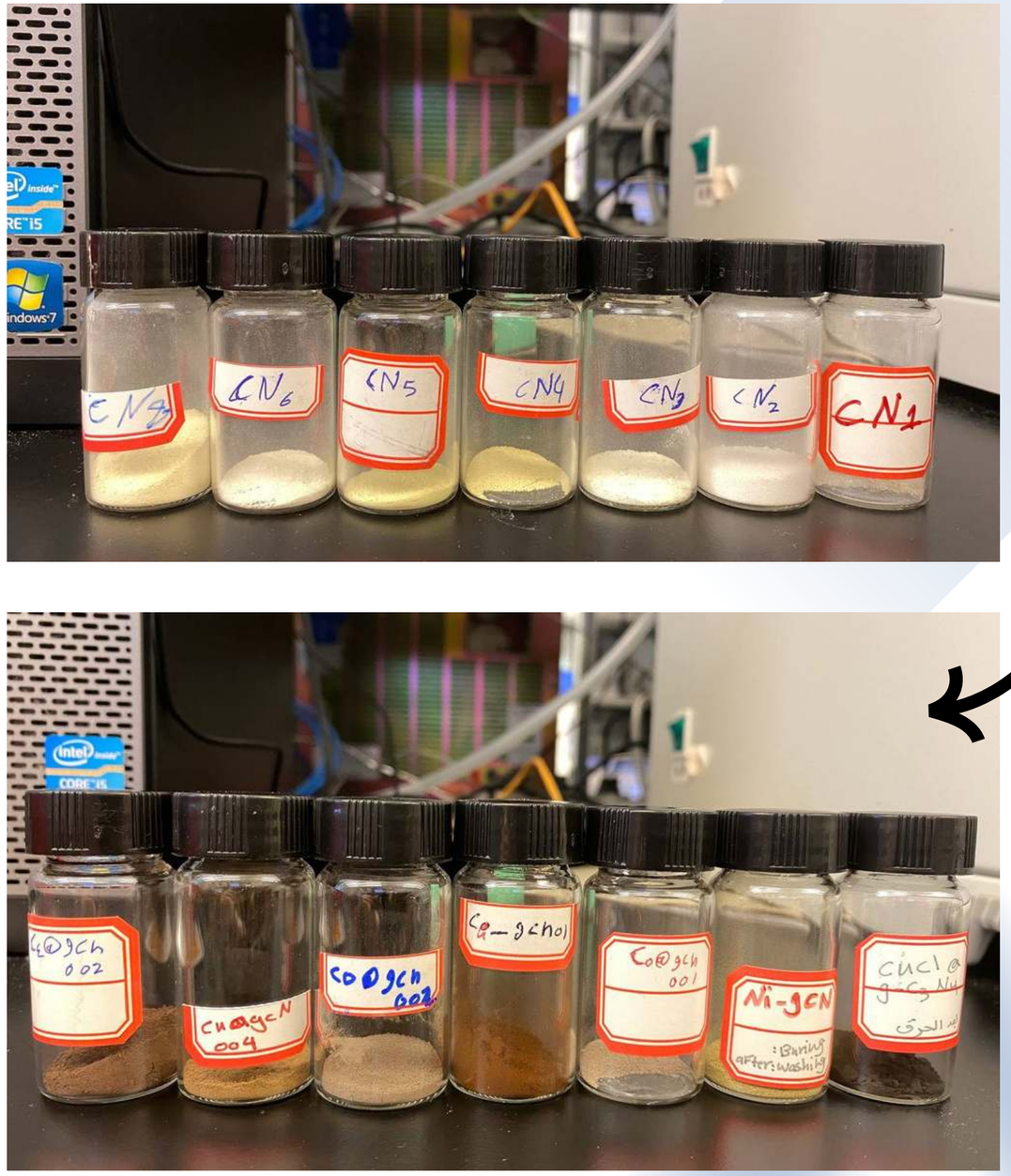




Different samples were placed in the furnace.
Each sample took one full day to finish.
Some samples were tested in air, and others were tested in nitrogen.
Different heating times and temperatures were used to study how they affect the samples.

Samples ($g-C_3N_4$) were synthesized at various temperatures.

Sample name	Preperation conditions				Notes
	Heating temp (C)	Heating rate (C/h)	Heating time (h)	Atmosphere	
CN1	450	4	5	air	
CN2	450	4	2 (7)	air	CN1 is further heated here
CN3	450	4	5	N2	
CN4	550	5	5	N2	
CN5	550	5	4	N2	
CN6	550	5	4	air	
CN7	650	5	4	air	The sample has completely evaporated
CN8	550	5	4	air	The sample was enclosed in aluminum foil



Characterization

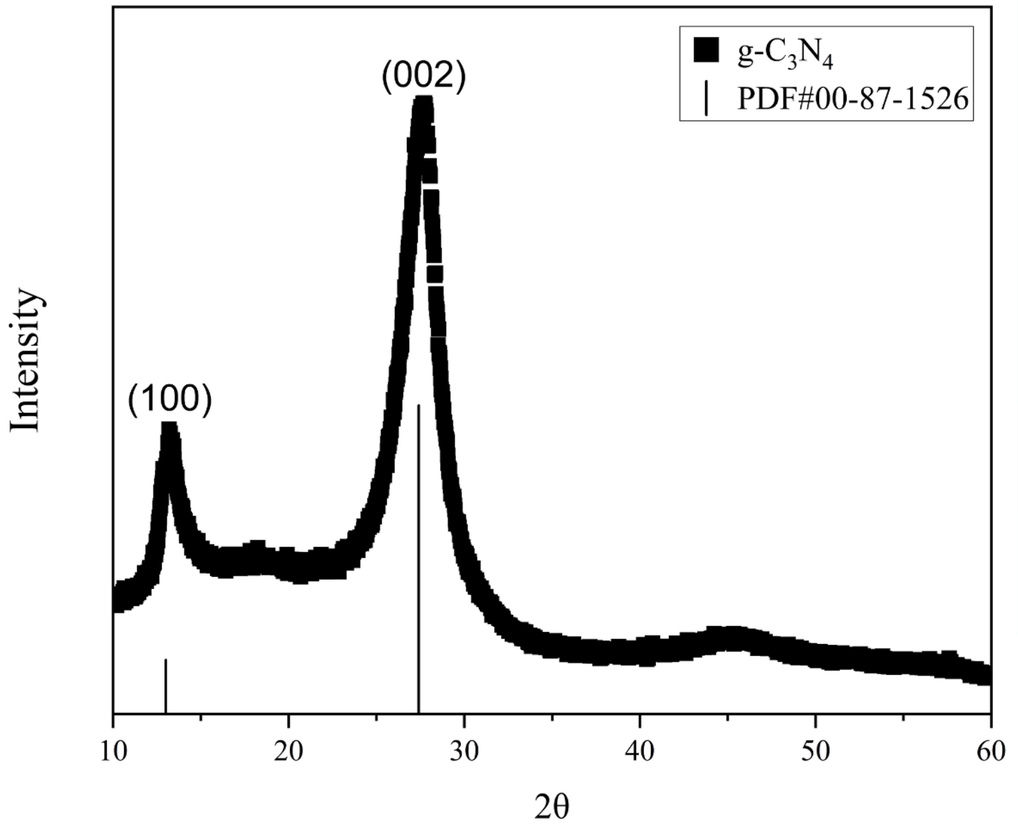
X-ray Diffraction analysis ,the diffraction pattern shows a strong peak at:

- 2θ = 27.58°

corresponding to the (002) crystal plane. shows a weaker peak at:

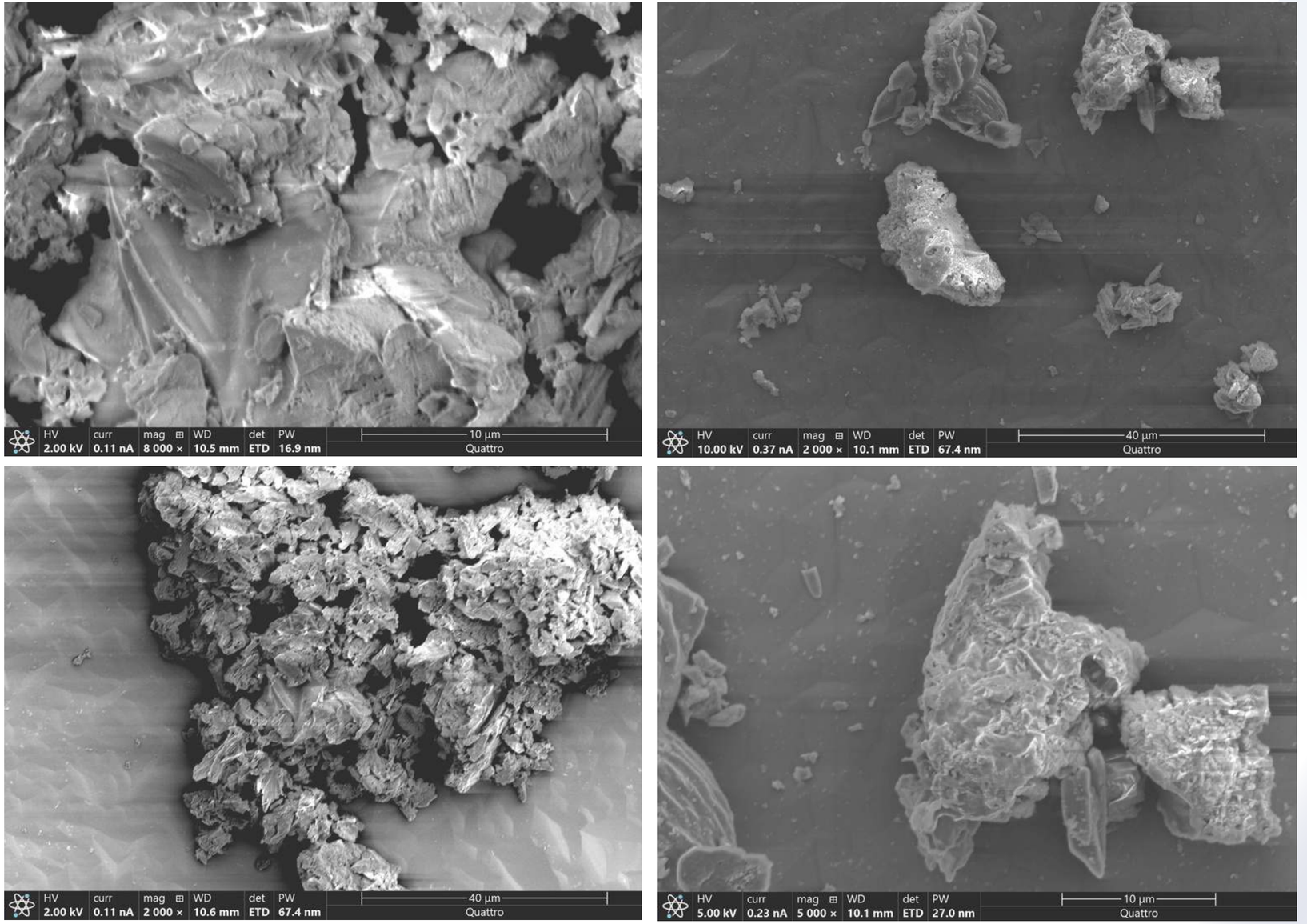
- 13°= (100) plane

both indicative of a layered graphitic carbon nitride (g-C_3N_4) structure.



XRD patterns of (g-C_3N_4)

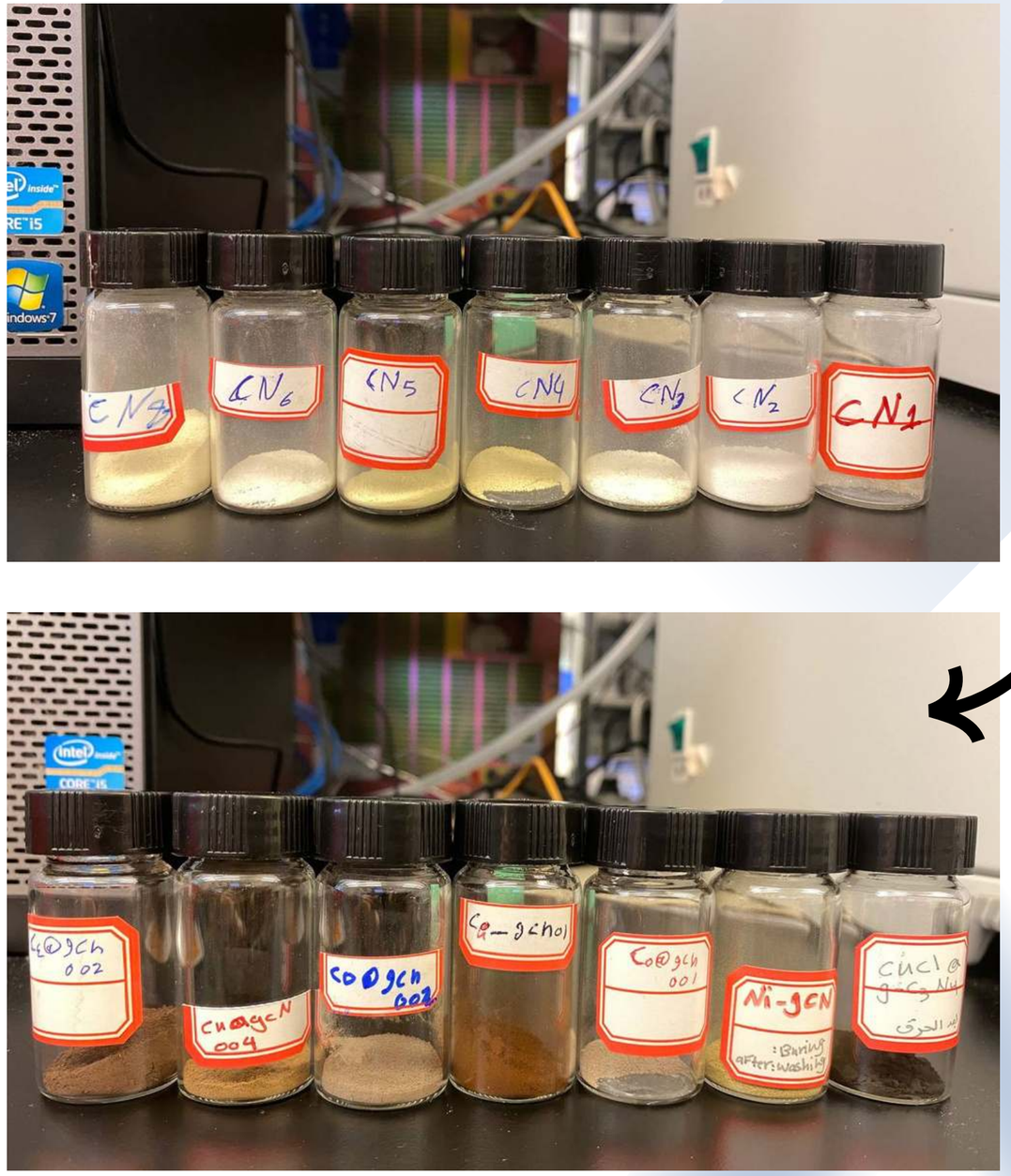
Characterization



Scanning electron micrographs of graphitic carbon nitride g–C_3N_4

Samples (g-C_3N_4) were synthesized at various temperatures.

Sample name	Preperation conditions				Notes
	Heating temp (C)	Heating rate (C/h)	Heating time (h)	Atmosphere	
CN1	450	4	5	air	
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CN4	550	5	5	N2	
CN5	550	5	4	N2	
CN6	550	5	4	air	
CN7	650	5	4	air	The sample has completely evaporated
CN8	550	5	4	air	The sample was enclosed in aluminum foil



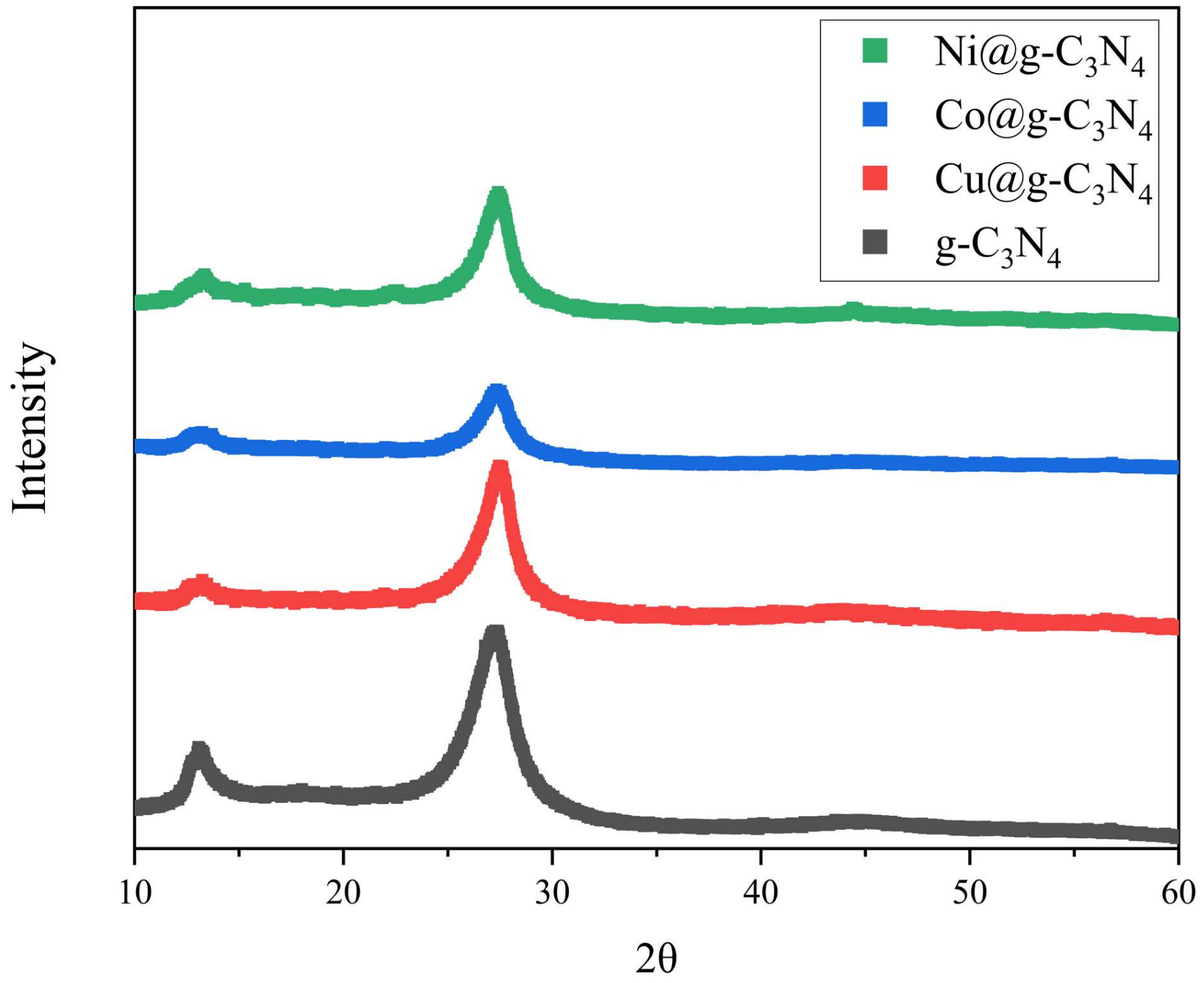
Doping Procedure

- **Solution Preparation:** Mix 500 mg melamine, 200 mg metal , 5 mL ethanol, and 5 mL deionized water in a 30 mL vial. Stir at room temperature for 24 hours.
- **Separation and Washing:** Centrifuge to collect the solid. Wash the solid three times with ethanol.
- **Drying:** Dry in a vacuum oven at 60°C for 24 hours. Grind into a fine powder.
- **Thermal Treatment:** Heat to 550°C under Nitrogen. Maintain for 4 hours. Cool under inert atmosphere.

As a result of the synthesis procedures, the following samples were successfully obtained:

- **Co_g-C_3N_4:** A cobalt-modified graphitic carbon nitride composite.
- **Cu_g-C_3N_4:** A copper-modified graphitic carbon nitride composite.
- **Ni_g-C_3N_4:** A Nickel-modified graphitic carbon nitride composite.

Characterization

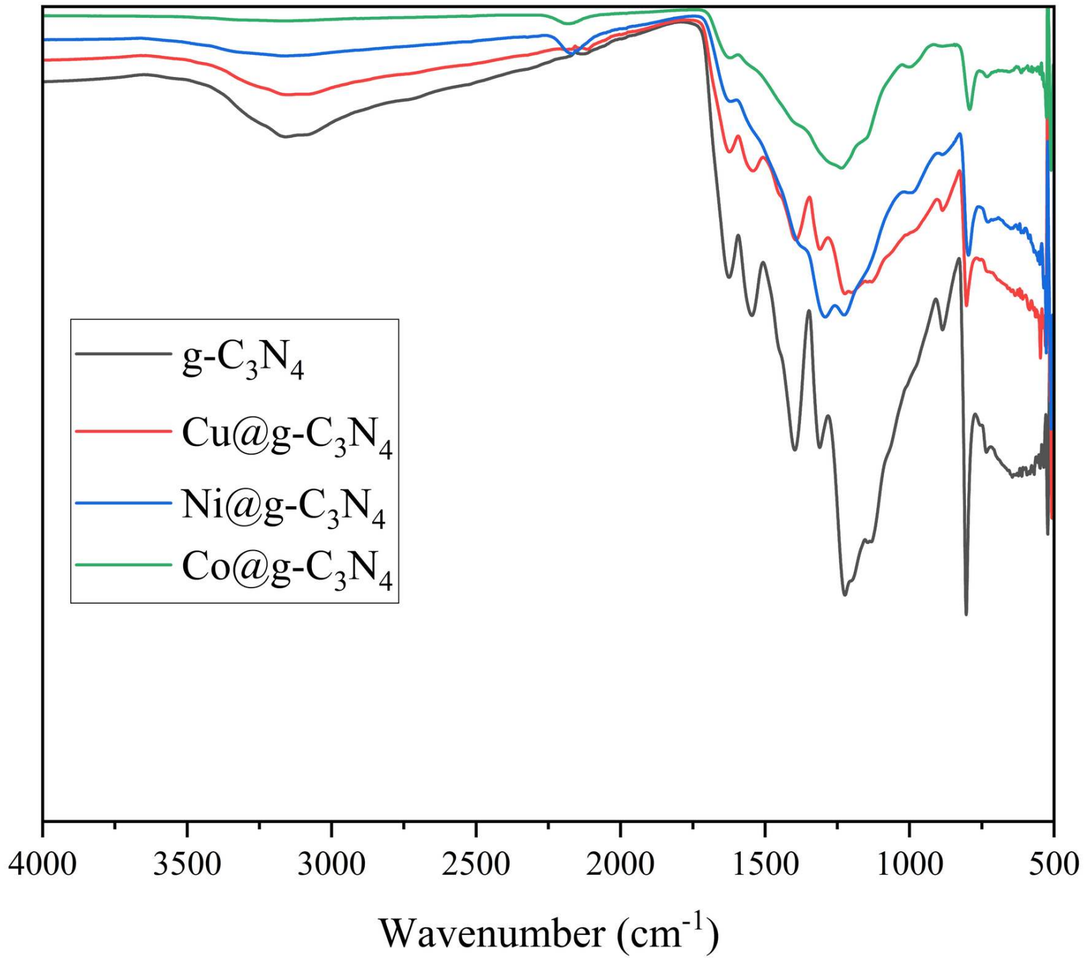


X-ray diffraction (XRD) pattern of g-C_3N_4-(Cu,Co,Ni)

Fourier Transform Infrared Spectroscopy (FTIR)

(FTIR) can be employed to analyze the functional groups, which help verify the carbon–nitrogen bonding. In general:

- Peaks around 800 cm^{-1} represent heptazine ring modes.
- Peaks near 1335 cm^{-1} and 1613 cm^{-1} correspond to C–N and C=N bonds, respectively .
- Peaks around 3340 cm^{-1} and 3460 cm^{-1} suggest the stretching and deformation modes of $-NH_2$ groups



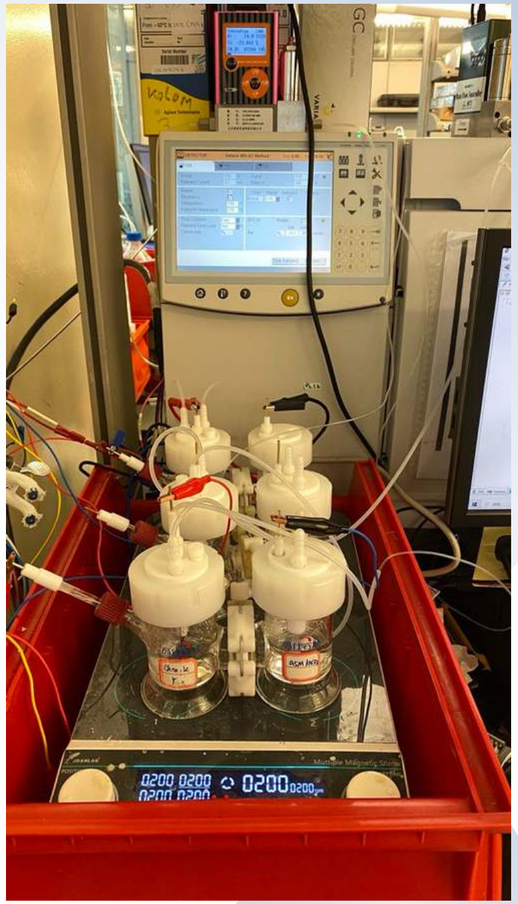
Electrochemical Concepts

- **Current potential curves** illustrate the relationship between electrical current and applied potential in electrochemical systems.

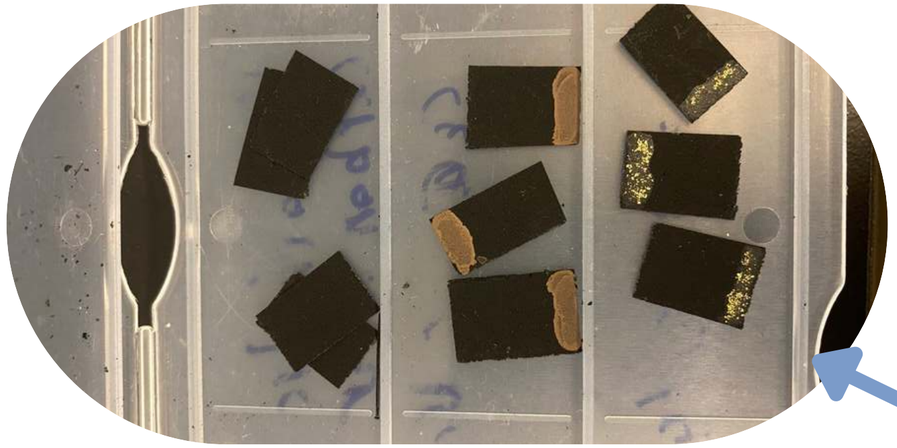
- **Current density** is a the amount of electrical current flowing per unit of electrode area.

- **Mass transport** is the movement of reactants, products, and ions between different components of the cell, such as the flow channels, gas diffusion layers.

- **Electric double layer (EDL)** describes the structure formed at the interface between an electrode (electronic conductor) and an electrolyte (ionic conductor).



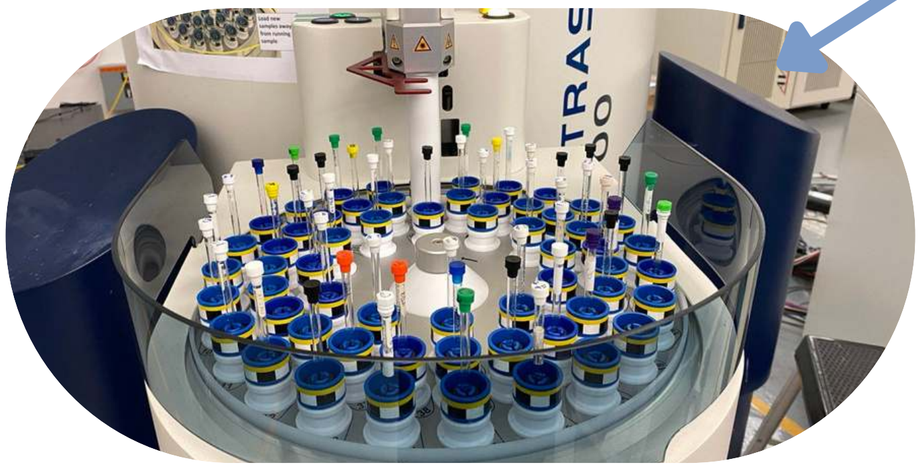
Electrochemical Setup



Carbon Paper Electrode



H-Cell

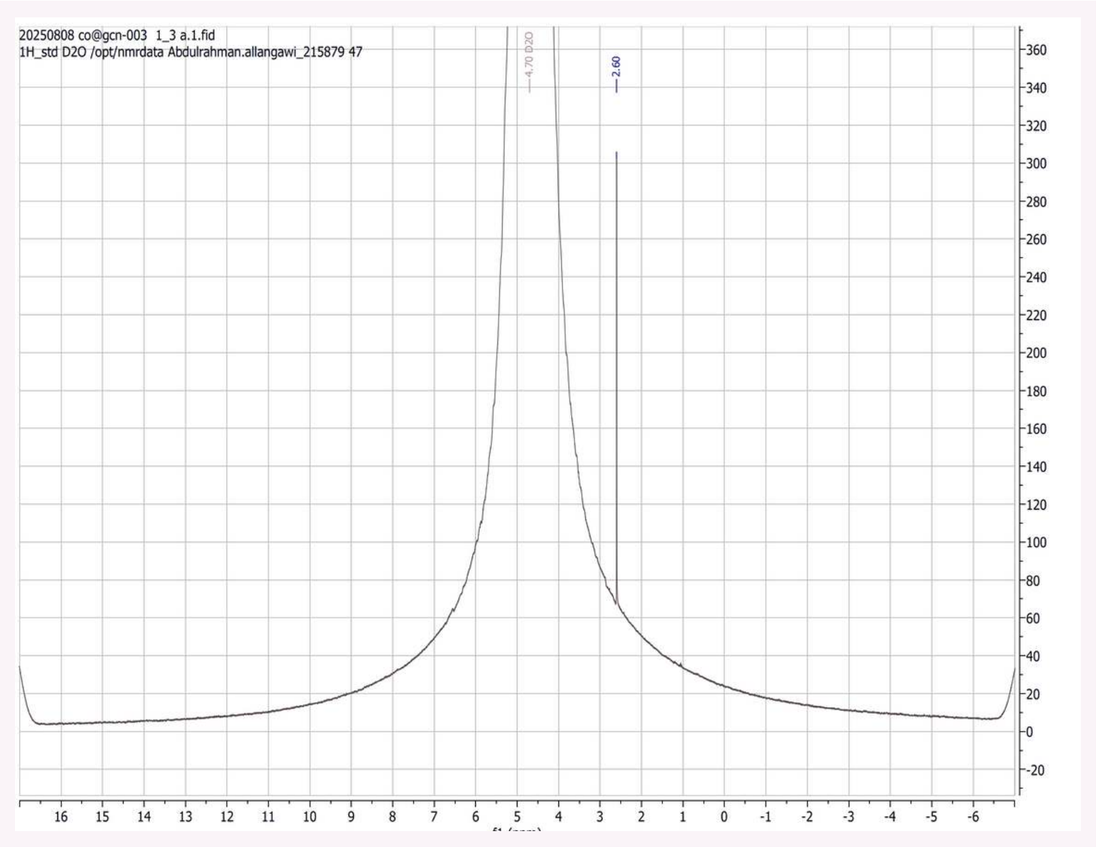


NMR

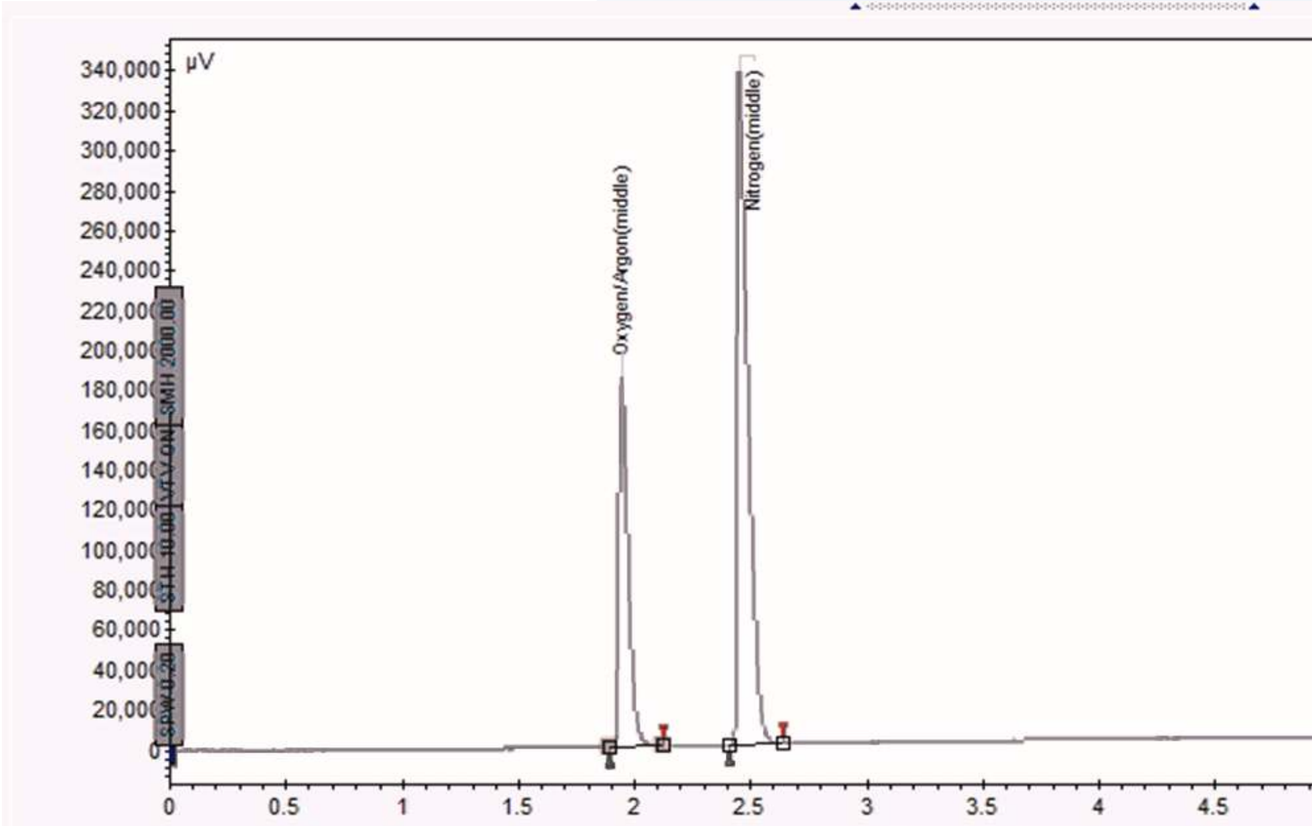


Gas Chromatography

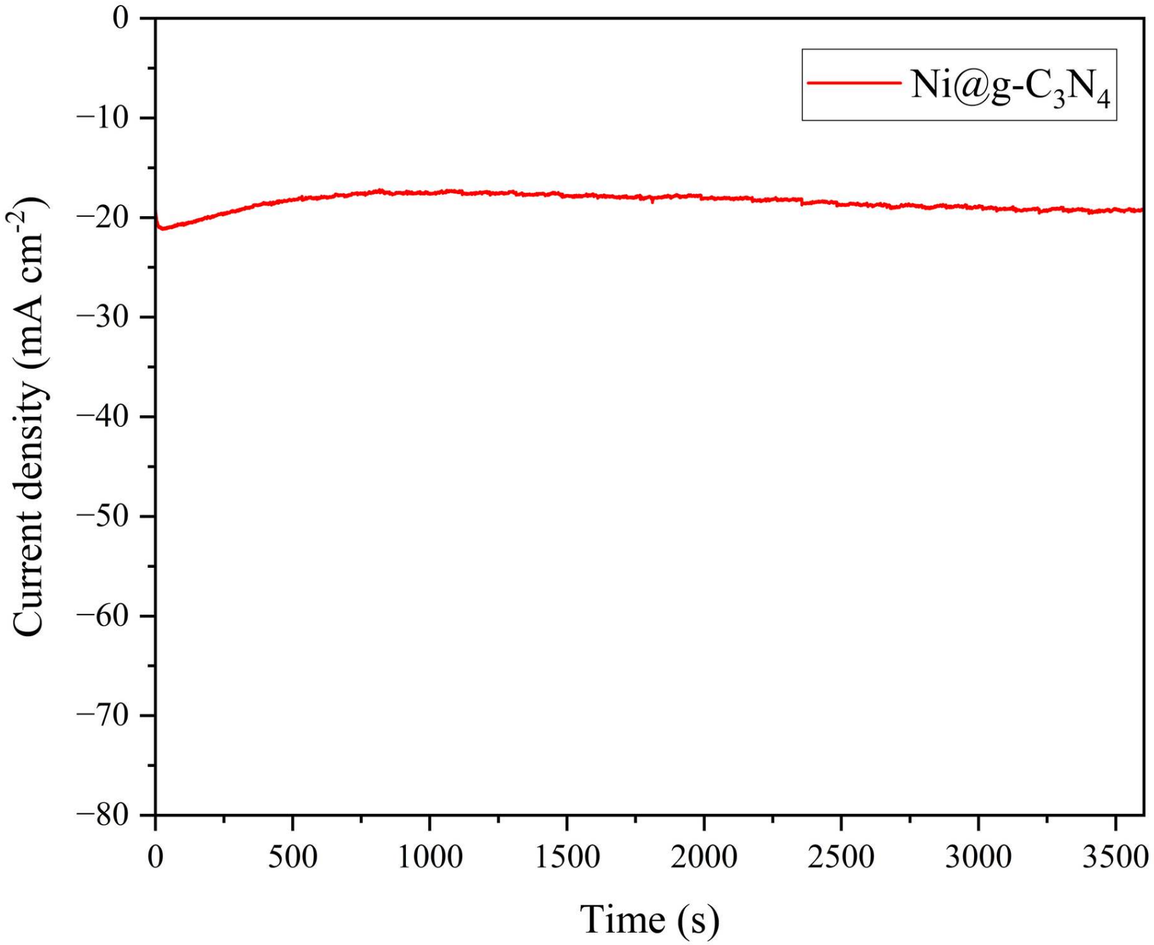
NMR


20250808 co@gcn-003 1_3 a.1.fid
1H_std D2O /opt/nmrdata Abdulrahman.allangawi_215879 47
4.70 D2O
2.60

GC


µV
Oxygen/Argon(middle)
Nitrogen(middle)

Chronoamperometry Curves (CA)



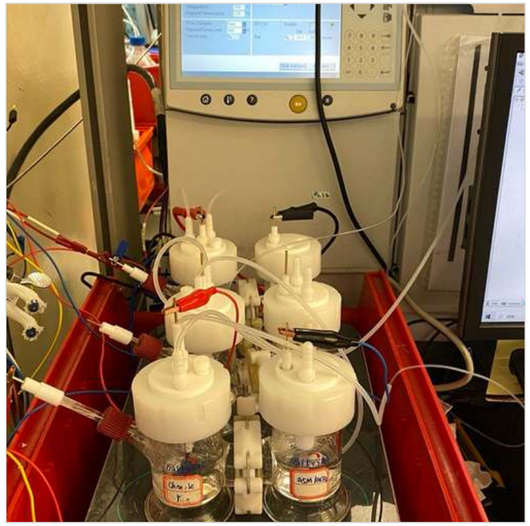
Lab Skills and Growth

- Learned how to find relevant scientific papers,
  - Extracted synthesis methods and experimental procedures from literature.
  - Improved scientific search and analysis skills.
  - Learned how to design and apply experimental approaches.

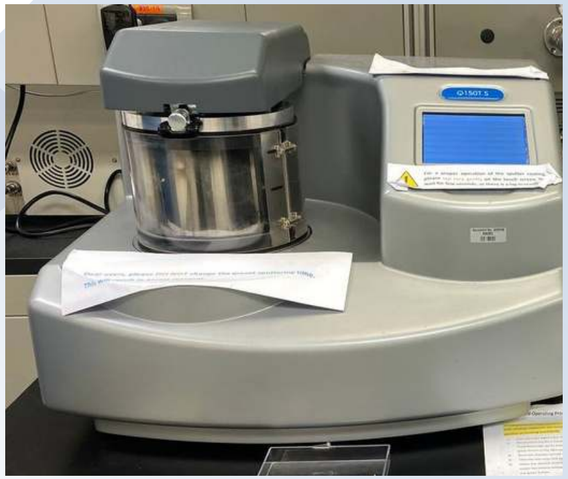
- I prepared for the experiment by learning how to write the steps clearly and in the correct order, which helped me approach the lab work with confidence and precision.
  - Developed strong lab report writing skills, including summarizing experiments, presenting results, and outlining next steps.
  - Applied theoretical knowledge to enhance practical execution and improve scientific documentation.

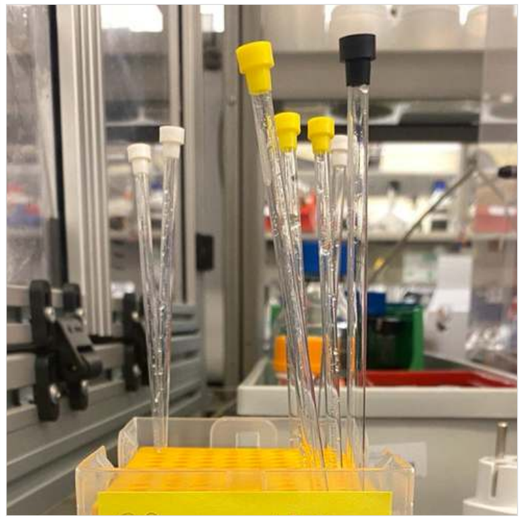
Laboratory experience:

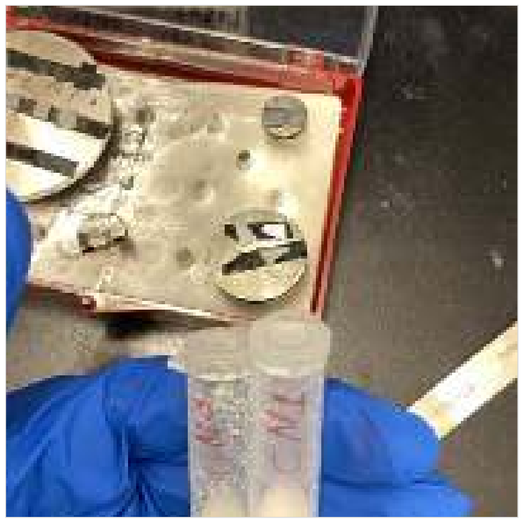
















Lab Skills and Growth

- Laboratory Instrumentation Experience

- Gained hands on experience with XRD, SEM, FTIR, and NMR.
- Learned how to properly set up instruments and resolve technical issues during operation.
- practical skills in adjusting tools and ensuring smooth experimental performance.

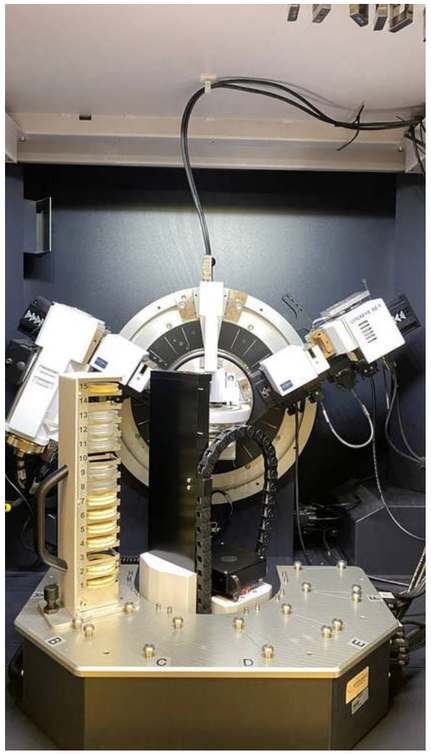
- Data Collection & Interpretation

- Improved ability to collect and interpret experimental data.
- Learned to identify key results and relate them to physical and chemical properties of materials.
- Developed skills in recognizing patterns and understanding the results.

I operated NMR, XRD, FTIR, and SEM instruments to analyze the structural, compositional, and morphological characteristics of the samples.



SEM



XRD



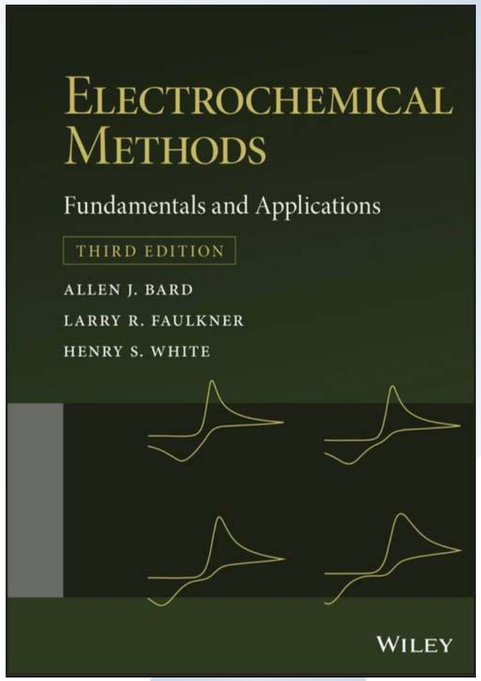
NMR



FTIR

Short term Plan – 9 Months

- I will complete a 6-month visiting student program to advance my skills in experimental techniques and analytical methods.
- Work with different catalysts to learn more about the electrolysis CO_2 reduction reaction and its real world applications.
- Assist in ongoing research projects and aim to be credited as a researcher in publications.
- Develop my understanding of basic science and fundamentals, while improving my scientific reading and literature review skills to stay updated with recent advances.
- Continue learning and applying Density Functional Theory (DFT) in my research.



Long Term Plan (5–7Years)

I intend to pursue master's and PhD studies as part of my long-term academic and professional development.

Materials i want to Explore During My Master’s to focus on electrocatalysis with emphasis on hydrogen evolution reaction (HER) and CO_2 reduction.

- I aspire to join Oxagon and contribute to national energy Projects.
- I want to represent Saudi Arabia at international energy conferences.
- I am committed to supporting girls in STEM through career empowerment in the energy field.





Thank You

Thank you for your attention.
Special thanks to Professor Huabin Zhang,PHD Students Abdulrahman, Miao ,and Changle for their support and guidance during my training.



First week of training at the Catalyst Center at King Abdullah University of Science and Technology (KAUST). KAUST arrival, office setup, and safety training.

Upon arrival, I began with safety training, where I attended a lab safety orientation, including lab safety, hazardous waste, and emergency preparedness. I completed the necessary training and earned a certificate of completion, which qualifies me to work at the Catalyst Center at KAUST.

On Thursday, I got ready to enter the lab and assist Jumanah Alharbi, M.S. Students on Professor Huabin Zhang's team, in preparing the necessary samples. I then put on my designated lab coat and safety goggles.

Lab Report: Preparation of an Organic Solution Using Acetic Acid

Name: Shahad Abdul Jabbar

1. Objective

The aim of this experiment is to prepare a solution containing acetic acid at a specific molarity and mix it with different organic solvents (such as acetone, THF, and DMF). Accurate calculations of the required amounts of acid and solvents were performed, followed by testing solubility and preparing the solution for further use.

2. Sample Preparation and Characterization Process:

A red solid sample was obtained after a 3-day heating process. The resulting material was washed three times using DMSO to remove any soluble impurities. Following the washing step, the sample was placed in a separation device for further purification. The final separation step will be conducted using the Cence H1850 centrifuge, which allows for efficient phase separation based on density differences**.**

3. Conclusion

This experiment highlights the importance of accurate calculations in solution preparation, especially when working with organic solvents like DMSO. A red solid sample was obtained after a 3-day heating process, indicating successful material transformation. The sample was washed three times with DMSO to remove residual soluble impurities, demonstrating the solvent's efficiency in purification. Following the washing step, the sample was placed in a separation device and then processed using the Cence H1850 centrifuge to isolate the solid phase based on density differences. Additionally, sonication was found to be effective in dissolving the solid components, proving to be a valuable tool in preparing and processing complex chemical mixtures. Overall, the combination of heating, washing, sonication, and centrifugation enabled efficient sample preparation for further analysis.





In the second week, we will follow a learning plan developed by the supervisors and Professor Huabin. On the first day, we started with Literature reading/experimental preparation.

The electrochemical reduction of CO_2 is a promising pathway for converting a greenhouse gas into useful fuels and chemicals. One key challenge is the competition from the hydrogen evolution reaction (HER), which reduces the selectivity toward CO_2RR. This experiment uses a synthesized catalyst to enhance CO_2 reduction and minimize H_2 production. Faradaic Efficiency (FE) and product analysis are used to evaluate catalyst performance.

Lab Report: Electrochemical CO_2 Reduction Using a Synthesized Catalyst.

Evaluation of Faradaic Efficiency for CO_2 Electroreduction Using a Synthesized Catalyst in $KHCO_3$ Solution.

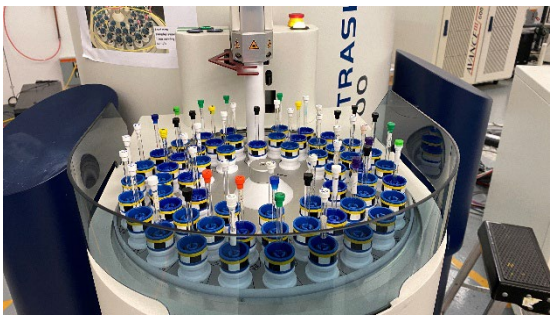
Objective:

To investigate the Faradaic Efficiency (FE) of a synthesized catalyst for the electrochemical reduction of carbon dioxide (CO_2RR), with the goal of suppressing hydrogen (H_2) evolution and enhancing CO_2 conversion under controlled potential conditions.

Materials and Chemicals:

- 0.5 M $KHCO_3$ (Potassium Bicarbonate) – Electrolyte
- Synthesized Catalyst – Loaded onto carbon paper (working electrode)
- Platinum foil – Counter electrode
- Ag/AgCl – Reference electrode
- Co2 Gas – Inert gas to purge oxygen
- Electrochemical H-type Cell – Divided cathode/anode chambers with membrane
- NMR Instrument – For post-reaction product analysis







Experimental Setup:

- A two-compartment H-type electrochemical cell was used.
- Working Electrode: Synthesized catalyst coated on carbon paper
- Counter Electrode: Platinum foil
- Reference Electrode: Ag/AgCl
- Electrolyte: 0.5 M $KHCO_3$ in both chambers
- Gas Control:Co2 was continuously bubbled to maintain an inert environment.
- Magnetic stirring was set at 200 rpm during the experiment.
- A potentiostat (BioLogic) was used to apply constant potential and collect data.

Experimental Conditions:

- Potential Range : −1.2 V TO −1.6V vs Ag/AgCl
- Duration (t_i): 10 minutes
- Data Sampling Interval (dta): Every 6 seconds
- Stirring Speed: 200 rpm
- Bandwidth: 1 (slow)
- Experiment Name: CA-$KHCO_3$-200rpm

Procedure:

Fill both sides of the H-cell with 0.5 M $KHCO_3$. Load the working electrode (carbon paper + catalyst) in the cathode side and platinum wire in the anode. Insert reference electrode near the working electrode. Purge both sides with Co2 for 15 minutes to remove O_2.Start the experiment by applying −1.400 V using the potentiostat. After completion, collect gas samples (if applicable) and liquid electrolyte. Analyze the liquid products using ^1H NMR spectroscopy. Calculate the Faradaic Efficiency for each product based on charge passed.

Observations:

- Visible bubble formation on the cathode side (gas evolution)
- Current was recorded continuously throughout the experiment
- No gas interference due to use of Co2
- The NMR spectrum may indicate the presence of formate products in the liquid phase.

Conclusion

Using a range of potentials between −1.2 V and −1.6 V, the synthesized catalyst demonstrated effective CO_2 electroreduction with suppressed hydrogen evolution. The ^1H NMR analysis confirmed the formation of formate and methanol, indicating successful conversion of CO_2 into valuable chemicals. The results highlight the potential of the catalyst for efficient and selective CO_2RR under mild conditions.

On the last day of the week, Professor Huabin Zhangmet with me again. The first meeting was on my first day in the office. Three other researchers from the professor's team, who were PhD students, joined the meeting: Abdulrahman Allangawi, Xiangyun (Tony) Xiao, and Miao Hu. The meeting was fruitful. The professor advised me to learn and work hard. He also suggested the possibility of joining the team and advised me to complete my master's degree to gain a better understanding of the field of hydrogen. I was able to present my CV to the professor. This week has come to a successful conclusion.

In the third week, we continue to follow the learning plan developed by the supervisors and Professor Huabin. During this week, the focus is on electrochemical applications, where we explore the theoretical foundations and engage in practical preparation and experimentation using appropriate techniques and equipment related to this field.

Lab Report: Synthesis of $CuCl_2/SnCl_2$/PVP-Based Catalyst for CO_2 Reduction

Date: 6-7July

1. Objective

The objective of this experiment is to synthesize a powder catalyst composed of copper chloride ($CuCl_2$), tin chloride ($SnCl_2$), and polyvinylpyrrolidone (PVP), to be used in the electrochemical reduction of carbon dioxide (CO_2). The synthesis aims to optimize loading mass, improve catalytic activity, and enhance the stability of the Cu–Sn-based electrocatalyst.

2. Materials and Reagents

- Copper(II) chloride ($CuCl_2$)
- Tin(II) chloride ($SnCl_2$)
- Polyvinylpyrrolidone (PVP)
- Deionized water (DI H_2O)
- Sodium hydroxide (NaOH), 2 M
- Centrifuge machine (F18)
- Oven



3. Procedure

1. Solution Preparation:
    - Dissolve 0.17 g of $CuCl_2$ in 100 mL of DI water
    - Add 40 mg of $SnCl_2$
    - Add 3.5 g of PVP
2. Mixing & Reaction:
    - Heat the solution at 55°C and stir for 30 minutes
    - Add 10 mL of 2 M NaOH solution
    - Stir the mixture at 51°C for 3 hours
3. Centrifugation & Washing:
    - The resulting mixture was centrifuged more than 3 times using a centrifuge machine (Model: F18) to wash the precipitate and
    - remove any unreacted materials.
4. Drying: The collected powder was placed in the oven at 120°C for 6 hours

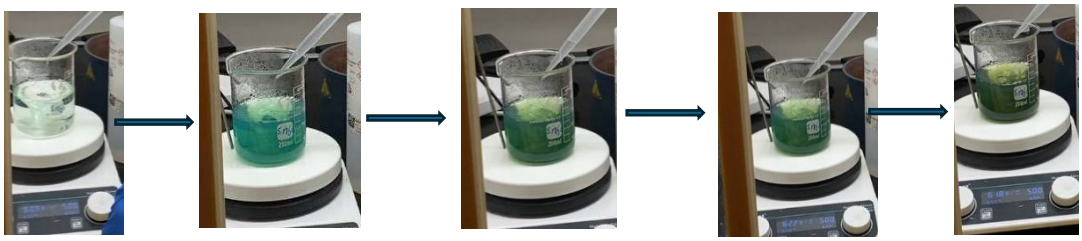
5. Post-treatment:

The final powder was subjected to heating at 200 °C for 1.5 hours to enhance its properties. After thermal treatment, the powder was collected, labeled, and briefly dried on carbon paper using a heat lamp. The carbon paper, provided as a demonstration product by Shengrong Company, was specifically chosen for its suitability as a catalyst support in electrochemical studies.

4. Observations

A colloidal suspension was formed upon the addition of PVP, followed by a noticeable change in the reaction mixture after the introduction of NaOH. The product was purified through repeated centrifugation and washing steps, then oven-dried to obtain a solid brownish powder. This powder underwent thermal treatment to complete the catalyst structure. The resulting dark-colored material will be evaluated for its activity and stability in future CO_2 electroreduction experiments. This solution-based synthesis method, combined with purification and thermal processing, provides a reliable approach for producing stable Cu–Sn electrocatalysts suitable for CO_2 conversion applications.

Solution preparation



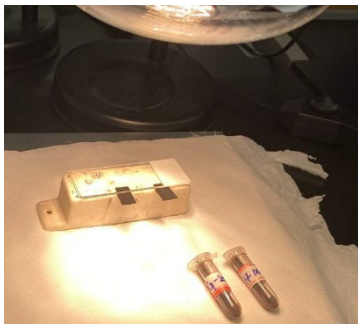
Devices used in the experiment:

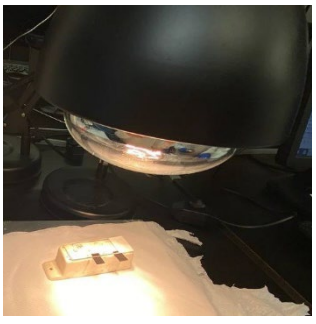
Centrifuge machine,**Tube furnace and Oven.**







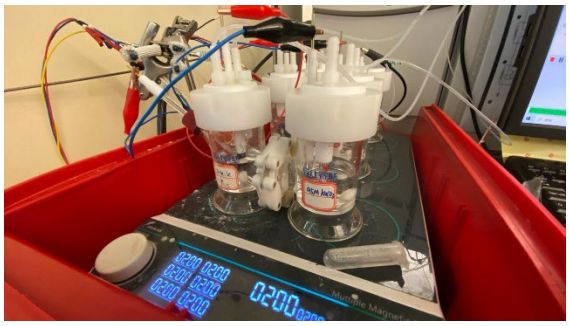


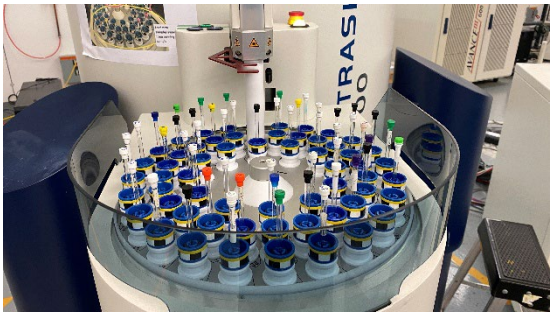


The catalyst was placed on carbon paper to test its performance in an electrochemical cell.

6.Electrochemical Measurements

After drying the synthesized Cu–Sn catalyst powder, we proceeded with the electrochemical testing to evaluate its CO_2 reduction performance. A new electrolyte solution was prepared with controlled pH, and CO_2 gas flow was initiated into the electrochemical cell. After a 10-minute stabilization step, we conducted chronoamperometry (CA) for 1 hour and 30 minutes to monitor the current over time under a constant potential. This was followed by linear sweep voltammetry (LSV) for 1 hour to observe the catalyst behavior across a range of potentials. Twenty minutes after starting the LSV, gas chromatography (GC) was initiated to analyze the evolved gaseous products. Additionally, after each potential step, we collected liquid samples from the cathode side for analysis by ^{1}H NMR, aiming to detect liquid reduction products such as formate. Finally, the electrolyte was replaced in preparation for the next round of experiments using a different potential or material sample.





Conclusion:

In this experiment, a Cu–Sn bimetallic catalyst was successfully synthesized using a wet chemical method involving $CuCl_2$, $SnCl_2$, and PVP as a stabilizing agent. The prepared catalyst underwent thermal treatment and drying steps, followed by careful washing and final drying on carbon paper to ensure purity and uniformity. The electrocatalytic performance of the catalyst for CO_2 reduction was then evaluated using electrochemical techniques including chronoamperometry (CA) and linear sweep voltammetry (LSV), with gas chromatography (GC) and ^{1}H NMR used to detect both gaseous and liquid products. The results highlight the importance of optimizing synthesis parameters and testing conditions to achieve stable and efficient catalytic activity. This experiment demonstrated not only the successful fabrication of a functional Cu–Sn catalyst, but also provided valuable insights into its behavior during electrochemical CO_2 reduction.

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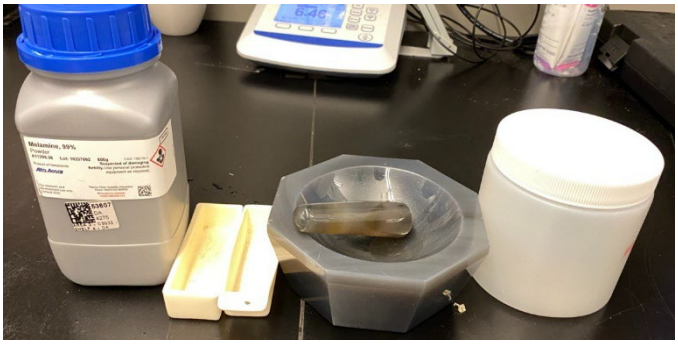
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Lab Report: Synthesis of g-C_3N_4 from Melamine

Date: 8-9 July

Introduction:

Graphitic carbon nitride (g-C_3N_4) is a metal-free, polymeric semiconductor material with a layered structure. It has gained significant attention due to its applications in photocatalysis, environmental remediation, and energy storage. One of the most common precursors for synthesizing g-C_3N_4 is melamine, which undergoes thermal polymerization at elevated temperatures.



Materials and Equipment:

- Melamine powder
- Mortar and pestle (or grinder)
- Tube furnace
- Ceramic crucible
- Heat-resistant gloves and safety goggles

Procedure:

- **Preparation of the Precursor:**
  - A suitable amount of melamine was weighed.
  - The melamine was ground thoroughly using a mortar and pestle to obtain a fine, uniform powder. This step ensures better thermal decomposition and uniformity in the final product.
- **Thermal Polymerization:**
  - The powdered melamine was transferred into a ceramic crucible.
  - The crucible was placed inside a tube furnace.
  - The furnace was programmed to heat gradually up to 450°C.
  - The temperature was maintained for 4 hours to allow complete thermal condensation and formation of g-C_3N_4.
  - After the heating process, the furnace was allowed to cool down naturally to room temperature.

Samples and Observations:

- Sample CN1:
  Melamine was heated at 450°C for 4 hours in air.
  Observation: The sample remained white with no visible change, indicating incomplete conversion to g-C_3N_4.
- Sample CN2:
  3 grams of CN1 were reheated at 450°C for 2 hours in air..
  Observation: No change in color was observed; the sample remained white, suggesting thermal stability and no further reaction.
- Sample CN3:
  A new batch of 4 grams of melamine was heated at 450°C for 4 hours under a nitrogen atmosphere.
  Observation: The sample turned light yellow, which indicates that we are on the right path toward synthesizing g-C_3N_4 under inert conditions.

Conclusion:

The experiment showed that heating melamine under nitrogen led to the formation of a yellow product, indicating progress toward g-C_3N_4 synthesis. In contrast, heating in air did not yield the desired result. However, factors like tube insulation may also influence the outcome, highlighting the need for careful control of thermal and environmental conditions.





Lab Report: Characterization of the samples obtained from Melamine

Date: 10 July

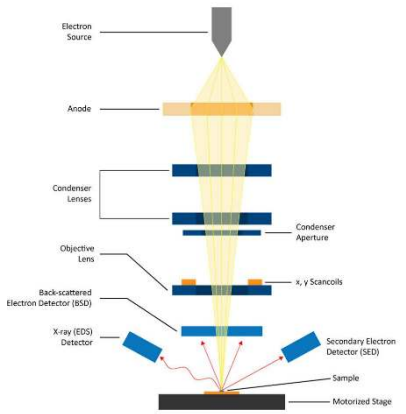
Characterization of Samples Using Scanning Electron Microscopy (SEM)After preparing the three samples, the next step was to perform morphological characterization (SEM). This step is essential for examining the surface structure and identifying the fine physical features of the samples.

What is Scanning Electron Microscopy?

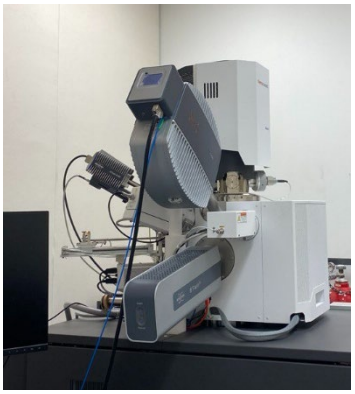
Scanning Electron Microscopy (SEM) is a powerful technique for obtaining high-resolution images of sample surfaces. It uses a focused beam of electrons to scan the surface, generating signals such as secondary electrons (SE), backscattered electrons (BSE), and X-rays, which provide information about surface topography and chemical composition.
SEM can achieve resolutions down to a few nanometers under optimal conditions, with a practical imaging range typically between ~50 nanometers and 100 micrometers.
It is widely used for analyzing solid materials, and insulating or biological samples may require special preparation, such as sputter coating or low-vacuum imaging, to minimize surface charging and beam damage.

How a Scanning Electron Microscope Works?

Electrons are produced at the top of the column in the electron gun and accelerated through the column at a specified accelerating voltage (1 keV – 30 keV). Condenser lenses and apertures act to reduce the beam diameter. The final lens in the column is the objective lens, which focuses the beam on the sample surface. The diameter of the beam in an SEM can range depending on the type of electron gun, accelerating voltage, and lens configuration. The sample itself is mounted on a stage in the chamber area and both the column and the chamber are maintained under vacuum by a combination of pumps. The level of the vacuum will depend on the design of the microscope. Some microscopes allow for variable pumping, allowing the sample chamber to be kept at a lower vacuum level than the rest of the column in order to support low-vacuum imaging.





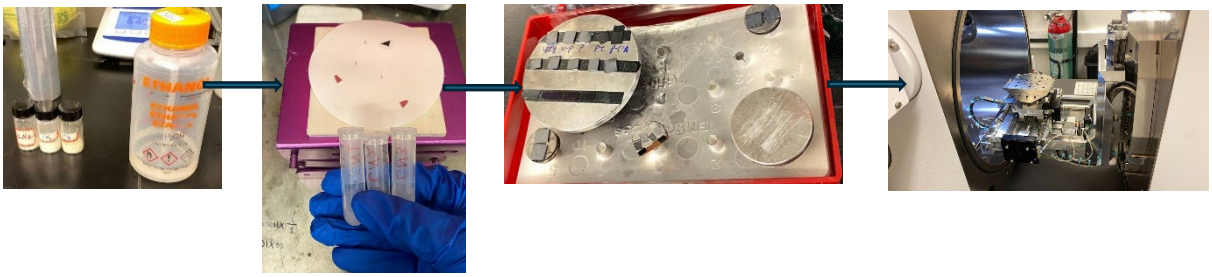


Sample Preparation for SEM Characterization

1. Solution Preparation:
- The samples were initially in powder form.
- Each sample was dissolved in an appropriate amount of ethanol in sealed containers.
- Complete dissolution of the samples in the solvent was ensured.

2. Slide Preparation:
- A small amount of the solution was dropped onto silicone slides using a micropipette.
- The slides were then mounted on a circular metal stub designed for SEM imaging.

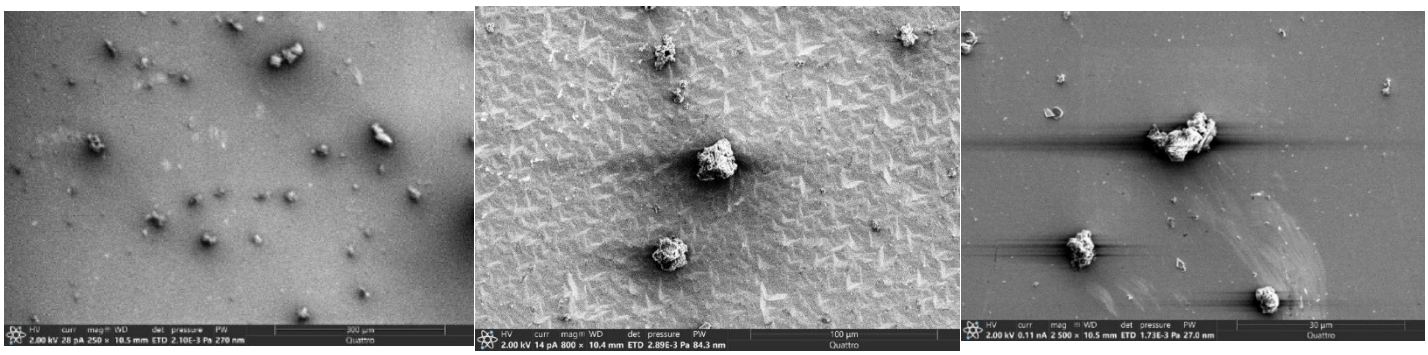
3. Loading into the SEM:
- The mounted samples were placed inside the SEM chamber to begin the imaging process.



SEM Imaging

Difficulties were encountered in obtaining clear SEM images due to the charging effect, which typically occurs with insulators and poorly conductive semiconductors. This issue was likely caused by the unsuccessful synthesis of g-C_3N_4. To resolve the charging problem, ultra-thin coatings of conductive materials, such as gold, can be applied to help dissipate the charge and enhance image quality.

From the SEM images that were obtained, a varied distribution of particles was observed across the sample surface, with noticeable differences in size and shape. Some particles were seen to be irregular, and certain areas showed greater accumulation than others. These features may be indicative of the material's nature or the conditions under which the sample was prepared. Overall, the images provided an initial indication of the surface characteristics, which could be further investigated to better understand the material's behavior in its intended applications.



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Lab Report: Synthesis of g-C_3N_4 from Melamine

On , this week Material characterization and DFT introduction.

We moved a sample (CN4) from the furnace that had been heated under nitrogen gas (N_2) at 550°C with a heating rate of 5°C/h for 5 hours. Right after that, a new sample (CN5) was placed in the furnace under the same nitrogen atmosphere and temperature, but for 4 hours.

We also prepared CN6, which was heated at 550°C for 4 hours in air. Another trial was done by heating a sample in air at 650°C for 4 hours, but the material completely evaporated, indicating it could not withstand such high temperature in air.

To improve the outcome, we lowered the temperature back to 550°C and wrapped the sample with aluminum foil before placing it in the furnace. We used 5 grams of the material. This adjustment was intended to protect the sample from oxidation and preserve its structure.

On the following day, we repeated the same process with the same conditions 5 grams of material wrapped in foil, heated at 550°C.

Earlier in the study, we had also prepared a set of samples under different heating conditions, as shown in the table below:

Sample name	Preperation conditions				Notes
	Heating temp (C)	Heating rate (C/h)	Heating time (h)	Atmosphere	
CN1	450	4	5	air	
CN2	450	4	2 (7)	air	CN1 is further heated here
CN3	450	4	5	N2	
CN4	550	5	5	N2	
CN5	550	5	4	N2	
CN6	550	5	4	air	
CN7	650	5	4	air	The sample has completely evaporated
CN8	550	5	4	air	The sample was enclosed in aluminum foil

Lab Report: Characterization of the samples obtained from Melamine

Characterization of Samples Using X-ray Diffraction (XRD),after preparing the samples, the next step was to perform structural characterization using X-ray Diffraction (XRD). This step is essential for identifying the crystalline phases present in the samples, determining their crystallinity, and analyzing the structural properties such as lattice parameters and crystallite size.

What is X-ray diffraction?

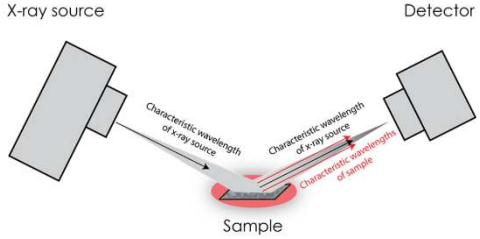
X-ray diffraction (XRD) is a versatile non-destructive analytical technique used to analyze physical properties such as phase composition, crystal structure and orientation of powder, solid and liquid samples. Many materials are made up of tiny crystallites. The chemical composition and structural type of these crystals is called their 'phase'. Materials can be single-phase or multiphase mixtures and may contain crystalline and non-crystalline components. In an X-ray diffractometer, different crystalline phases give different diffraction patterns. Phase identification can be performed by comparing X-ray diffraction patterns obtained from unknown samples to patterns in reference databases. This process is like matching fingerprints in a crime scene investigation. The most comprehensive compound database is maintained by ICDD (International Center of Diffraction Data). You can also build a reference database from measured pure-phase diffraction patterns, from patterns published in the scientific literature, or from your own measurements. The relative strengths of the patterns from different phases in a multiphase mixture are used to determine the full composition of a sample.

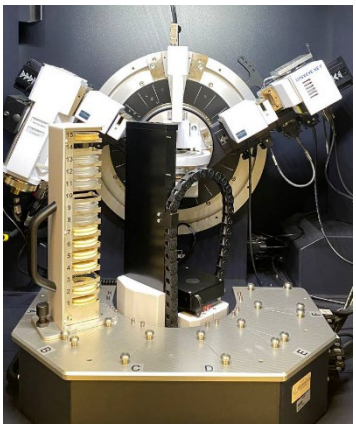
How Does X-ray Diffraction (XRD) Instrumentation Work?

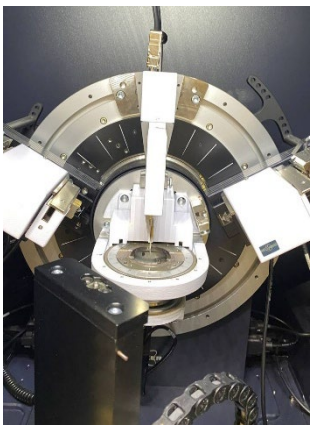
An X-ray diffraction (XRD) instrument consists of three main components:

- X-ray source
- Sample holder
- XRD detector

The X-rays generated by the source illuminate the sample. These rays are then diffracted by the crystalline phases within the sample and directed toward the detector. By adjusting the position of the X-ray tube, the sample, or the detector to vary the diffraction angle (2θ — the angle between the incident and diffracted beams), the intensity of the diffracted rays is measured and recorded. Depending on the geometry of the diffractometer and the type of sample, the angle between the incident beam and the sample can be either fixed or variable, and it is typically paired with the angle of the diffracted beam.







Sample Preparation for XRD Characterization:

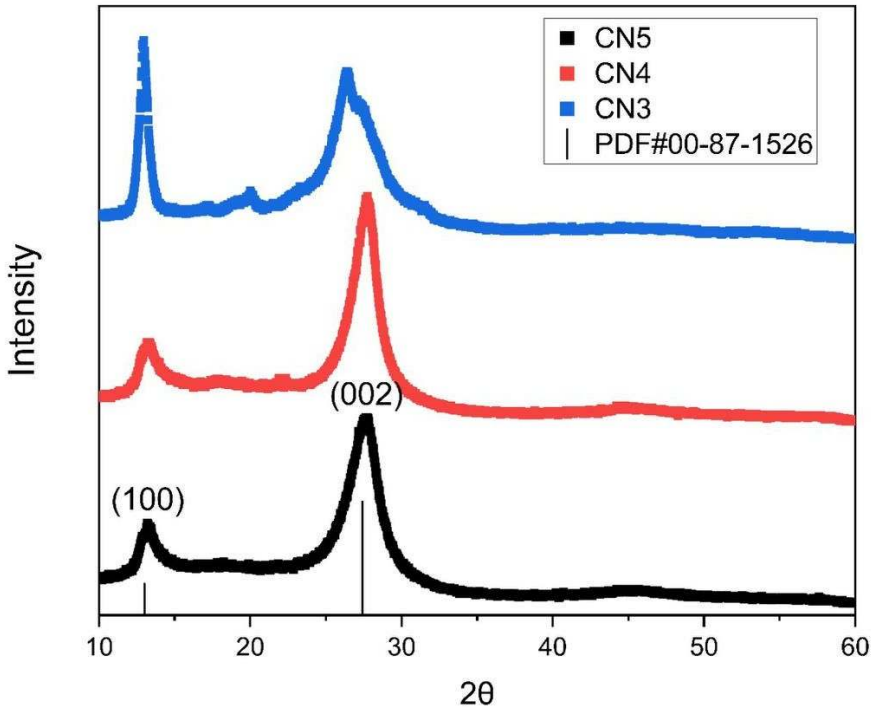
To prepare samples for X-ray diffraction (XRD), the material is first finely ground to ensure uniform particle size and reduce preferred orientation effects. The powder is then dried if necessary and carefully mounted on a sample holder, ensuring a flat and even surface. Proper packing is essential to avoid texture or strain. This preparation ensures accurate diffraction patterns and reliable phase identification.





XRD Lab Report:

We analyze the crystal structure and phase purity of CN3, CN4, and CN5 samples using X-ray diffraction.



Results and Observations:

Two main diffraction peaks were observed in all samples:

- Around **13.1° (2θ)**: (100) plane – in-plane repeating units.
- Around **27.4° (2θ)**: (002) plane – interlayer stacking.

CN5 (Black): Strong and sharp (002) peak, visible (100) peak

CN4 (Red): Clear (002) peak, weaker (100) .
CN3 (Blue): Broad (002) peak, weak (100) .

Conclusion:

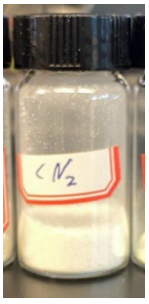
X-ray diffraction (XRD) patterns confirm the successful formation of graphitic carbon nitride (g-C_3N_4) across all synthesized samples. Among them, CN5 exhibited the highest crystallinity, indicating superior layer stacking and a more ordered structure—likely a result of optimized synthesis parameters such as temperature and duration. In contrast, crystallinity progressively declined from CN5 to CN3, which may be attributed to shorter heating times or suboptimal reaction conditions. The clear identification of g-C_3N_4's crystal structure through XRD highlights the effectiveness of the synthesis approach and the structural integrity of the resulting materials.

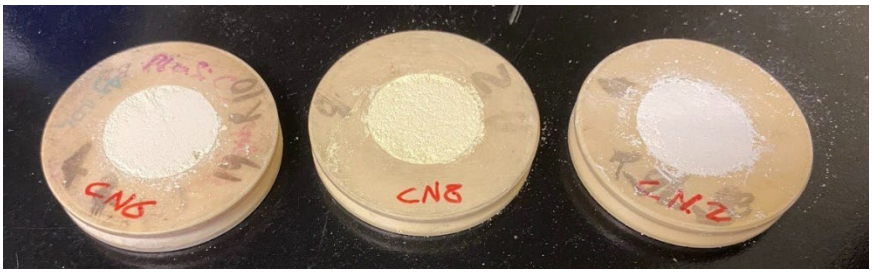
Sample Preparation for XRD Characterization:

The other samples—**CN2, CN6, and CN8**—have been prepared for subsequent XRD analysis to evaluate their crystal structure and compare them with the previously analyzed samples. This will help in understanding the impact of different synthesis conditions on the quality of the resulting material.









Lab Report: Synthesis of Cu-N4 Single Atom Catalyst (SAC)

. Objective:

The aim of this experiment is to synthesize a copper-based single atom catalyst (Cu-N4 SAC) using a wet-chemical method followed by pyrolysis.

Materials and Chemicals:

- Melamine (500 mg)
- Copper(II) chloride ($CuCl_2$) (200 mg)
- Ethanol (5 mL)
- Distilled water (5 mL)
- 30 mL glass vial
- Vacuum oven
- Centrifuge
- Mortar and pestle or grinder
- Tube furnace
- H_2/Ar gas mixture (5 vol.% H_2 in Ar)

Procedure:

1.Solution Preparation:

Add 500 mg of melamine, 200 mg of $CuCl_2$, 5 mL of ethanol, and 5 mL of deionized water into a 30 mL vial.

Stir the mixture continuously for **24 hours** at room temperature.

2. Separation and Washing

After synthesis, the solid product was collected by centrifugation.

The collected material was washed three times with ethanol to remove residual precursors and impurities:

First wash with 20 mL of ethanol

Second wash with 15 mL of ethanol

Third wash with 10 mL of ethanol

3. Drying

The washed samples were placed in an oven and dried at 60°C.

After drying, the samples were ground thoroughly into a fine powder to ensure uniformity.

4. Thermal Treatment

The powdered samples were then subjected to thermal treatment in a furnace under a nitrogen (N_2) atmosphere.

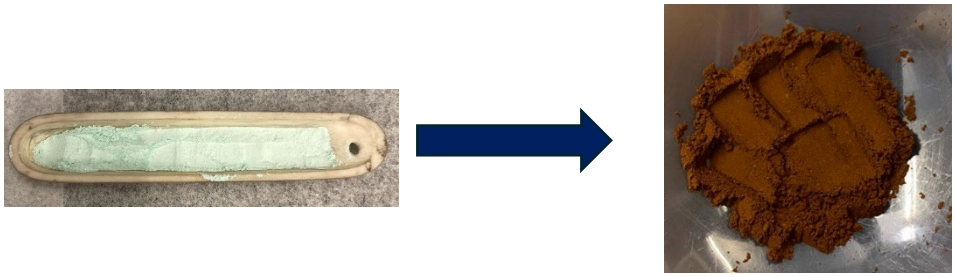
This step was performed to enhance structural stability and remove any remaining organic residues.

5. Characterization

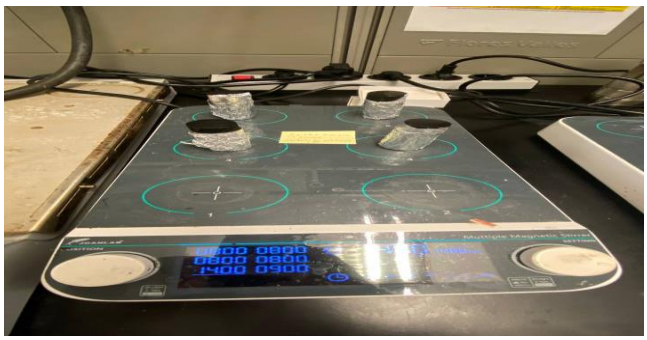
Following thermal treatment, the samples were analyzed using X-ray diffraction (XRD) to confirm the formation of g-C_3N_4 and evaluate their crystallinity.



AFTER BURING:



This week, I repeated the full synthesis procedure in order to verify the reproducibility of the experiment and to prepare additional material for analysis. I followed the same steps as before, including the mixing, washing, drying, and thermal treatment stages.
In addition to using **Copper(II) chloride ($CuCl_2$)** as in the original procedure, I also carried out a **second synthesis using Copper(I) chloride (CuCl)** under identical experimental conditions. In the preparation of CuCl samples, each specimen was purged with nitrogen gas for one minute to eliminate reactive oxygen species. Subsequently, the samples were sealed in aluminum foil to prevent the oxidation of CuCl into $CuCl_2$ .The purpose of this comparison is to evaluate whether the oxidation state of the copper source affects the final structure, color, or other physical properties of the synthesized product. Both sets of samples were successfully prepared and are now ready for further characterization and analysis.





Sample Preparation and Characterization:
Additionally, after completing the thermal treatment in the **tube furnace** under a **nitrogen (N_2) atmosphere**, the final products were collected and processed. As a result of the synthesis procedures, the following samples were successfully obtained:

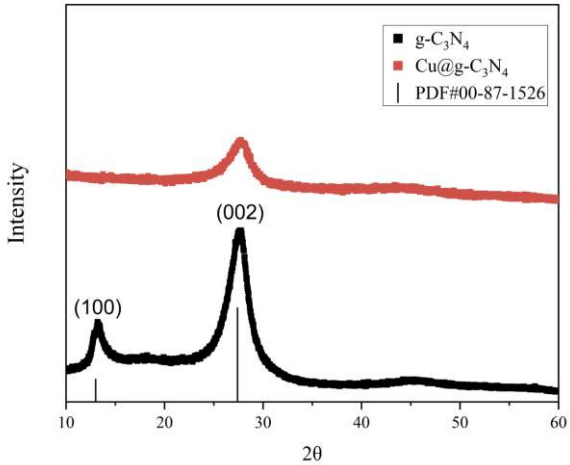
- **Co_g-C_3N_4**: A cobalt-modified graphitic carbon nitride composite.
- **Cu_g-C_3N_4**: A copper-modified graphitic carbon nitride composite.

Both samples were collected as fine powders after cooling and thorough grinding. To investigate their structural features and confirm the successful integration of the metal species into the carbon nitride framework, the samples were characterized using **X-ray diffraction (XRD)**. The XRD analysis provided insights into the crystallinity and phase composition of the synthesized materials.

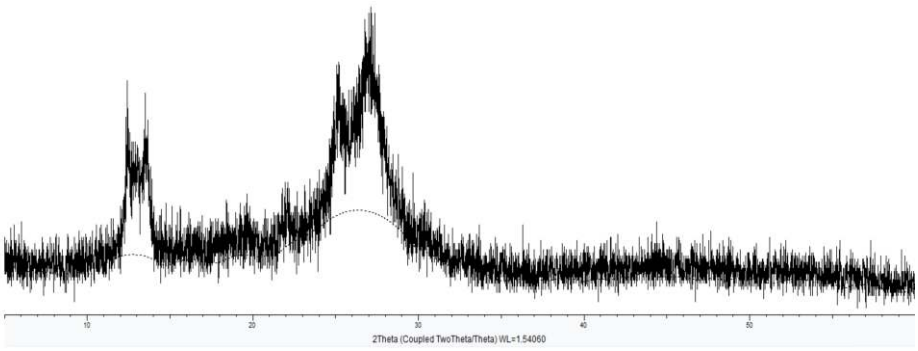


X-ray diffraction (XRD) analysis was performed to investigate the structural profile of the prepared samples. The resulting patterns are presented below and provide insight into the crystalline characteristics based on diffraction behavior.

The XRD pattern of the **Cu@g-C_3N_4** sample displays crystalline features that may be associated with a g-C_3N_4-like phase, notably a prominent peak around $2\theta \approx 27°$. Such structural organization could potentially influence charge transport within the electrocatalytic system. The inclusion of copper into the framework might play a role in activating certain sites or modulating electronic behavior relevant to CO_2 reduction reactions, particularly towards methanol formation. While the exact correlation between structure and catalytic performance remains to be fully understood, the observed characteristics suggest promising directions for further investigation.



The (XRD) pattern of **Co@g-C_3N_4** reveals a distinct peak at $2\theta \approx 27°$, indicating a crystalline g-C_3N_4 phase with a well-ordered structure. This structural integrity is beneficial for facilitating charge transport in electrocatalytic systems. The incorporation of cobalt (Co) enhances the active sites and electronic conductivity, making the material especially promising for electrochemical CO_2 reduction to methanol (CH_3OH). Methanol production requires multi-electron transfer steps and precise adsorption control, both of which can be supported by the Co-modified g-C_3N_4 framework. The synergy between cobalt and g-C_3N_4 may contribute to higher selectivity and Faradaic efficiency for CH_3OH formation under optimal conditions.



Electrochemical Testing of CO_2 Reduction Catalyst :Lab Report

Objective

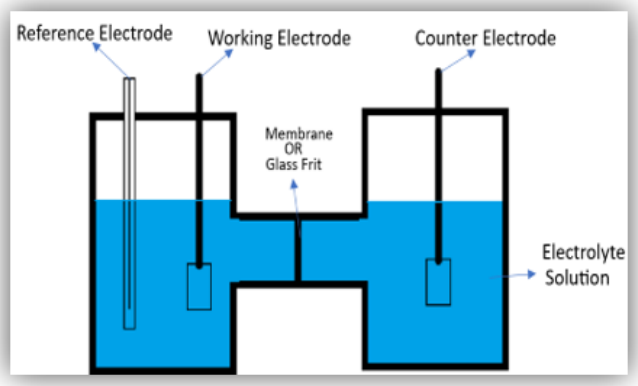
The objective of this experiment is to evaluate the catalytic activity and product formation efficiency of a coated electrode for CO_2 electroreduction. This was achieved through electrochemical techniques in a controlled H-cell setup.

Catalyst was carefully and uniformly loaded onto carbon paper before measurements. Accurate calibration of the reference electrode was essential for precise conversion to RHE. CO_2 gas bubbling and consistent stirring were maintained throughout to ensure reproducibility under simulated reduction conditions.

Materials and Apparatus:

- **Working Electrode:** Platinum (Pt)
- **Counter Electrode:** Platinum (Pt)
- **Reference Electrode:** Ag/AgCl
- **Electrolyte:** 0.5 M $KHCO_3$,50 ml solution with 5 used in both compartments
- **Stirring:** Stir bars placed in both cell compartments
- **Gas Flow:** Continuous bubbling of CO_2 in the compartments
- **Reference Electrode Calibration:**

$$E\{RHE\} = E\{Ag/AgCl\} + 0.197\ V = 0.493\ V\ \{at\ pH\ 7\}$$



Experimental Procedure:

1. **System Preparation:**

Both compartments of the H-cell were filled with $KHCO_3$ solution and stirred to maintain homogeneity. The cell was sealed tightly to sustain a pure CO_2 atmosphere.

2. **Catalyst Activation and Chronoamperometry (CA):**

An initial CA measurement was performed at –0.95 V vs RHE (equivalent to –1.645 V vs Ag/AgCl) for 10 minutes to activate the catalyst surface. Subsequent CA measurements were carried out at –1.5 V, –1.3 V, and –1.1 V vs Ag/AgCl for 1 hour. During this phase, liquid samples were collected at 15, 30, and 45 minutes and analyzed later using ^1H NMR to detect liquid-phase products.

Catalyst was carefully and uniformly loaded onto carbon paper before measurements. Accurate calibration of the reference electrode was essential for precise conversion to RHE. CO_2 gas bubbling and consistent stirring were maintained throughout to ensure reproducibility under simulated reduction conditions.

The catalyst was placed on carbon paper to test its performance in an electrochemical cell.







Lab Report: Preparation of LnNi-PHI Catalysts

To synthesize a crystalline photocatalyst carrier based on potassium poly(heptazine imide) (K-PHI), incorporate bipyridyl-based nickel complexes via cation exchange, and evaluate the resulting heterogeneous LnNi-PHI catalysts in visible-light-driven C-heteroatom cross-coupling reactions.

Materials & Methods

1. Preparation of K-PHI Photocatalyst

- **Reagents:**
  - Melamine: 1.0 g
  - NH_4Cl: 3.0 g
  - KCl: 10.0 g
- **Procedure:**
  - The mixture was finely ground and subjected to thermal polymerization at 550 °C (2.3 °C/min ramp rate) for 4 hours under nitrogen flow .
  - After natural cooling, the yellow solid was washed 3 mL times with deionized (DI) water, centrifuged, and then dried under vacuum overnight at 60 °C.
- **Preparation of N:-PHI**
- **Reagents:**
  - K-PH: 200 mg
  - Ni-bpy: 40 mg
  - KCl: 10.0 g
  - DMF (N,N-Dimethylformamide): 5 mL
- **Procedure:**

K-PHI and Ni-bpy were placed in a clean flask, followed by the addition of DMF to form a suspension. Nitrogen gas was used to purge oxygen and create an inert atmosphere. Once sealed, a magnetic stir bar was added, and the mixture was stirred at room temperature for three days to allow the reaction to proceed.

After the stirring period, the resulting solid was collected and washed thoroughly in the following sequence:
• Two times with DMF to remove unreacted organic species.
• Two times with deionized water to eliminate water-soluble impurities.
• Two times with MeCN (acetonitrile) to remove any remaining solvent residues.

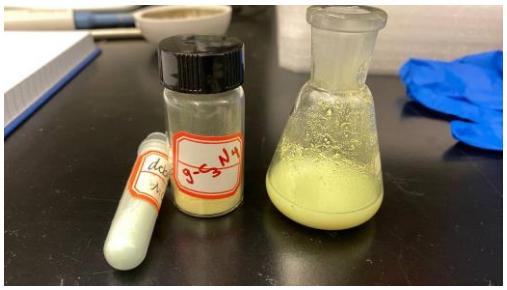
Finally, the purified material was dried in a vacuum oven until fully dry.

Gradual color change was observed during the stirring process, indicating the progress of the reaction. The washing steps yielded a cleaner product, free from visible residues, and the final material was collected as a dry powder after vacuum drying.

Conclusion:

The N:-PHI compound was successfully synthesized through a nitrogen-purged reaction and continuous stirring for 3 days. The sample underwent a multi-step washing process and was dried under vacuum, resulting in a purified solid ready for further analysis.







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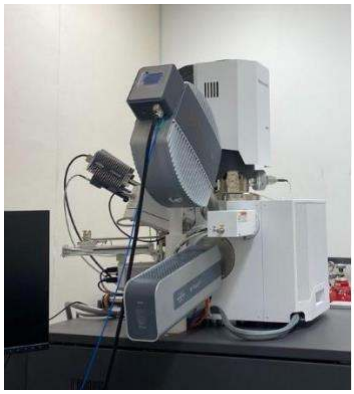
Rport: Characterization of the samples

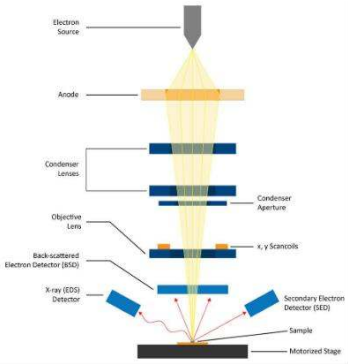
{CN5,CO@g-C3N4 and Cu@g-C3N}

Characterization of Samples Using Scanning Electron Microscopy (SEM)After preparing the three samples, the next step was to perform morphological characterization (SEM). This step is essential for examining the surface structure and identifying the fine physical features of the samples.

Scanning Electron Microscopy (SEM)

is a technique that captures detailed images of solid surfaces using a focused beam of electrons. As the electrons interact with the sample, they produce signals that reveal surface texture and chemical features. The electrons originate from an electron gun and pass through a column of lenses before striking the sample, which is placed in a vacuum chamber to reduce interference. For delicate materials like biological or insulating samples, adjustments such as surface coating or reduced vacuum may be used to prevent damage. SEM is widely applied in fields that require precise structural and material analysis at a microscopic scal.





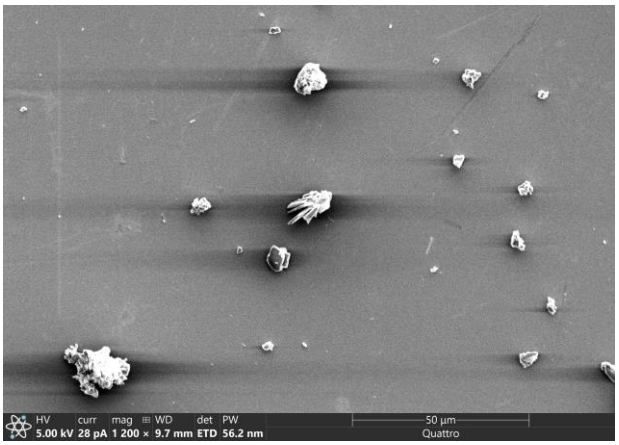
Sample Preparation for SEM Characterization

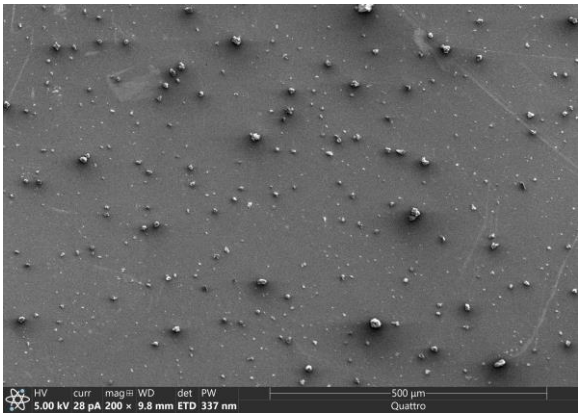
The samples, initially in powder form, were fully dissolved in appropriate volumes of ethanol within sealed containers to ensure complete solubilization. A small portion of each solution was then carefully deposited onto silicon slides using a micropipette. These prepared slides were subsequently affixed to circular metal

stubs specifically designed for SEM imaging. Finally, the mounted samples were loaded into the SEM chamber to initiate the imaging process.

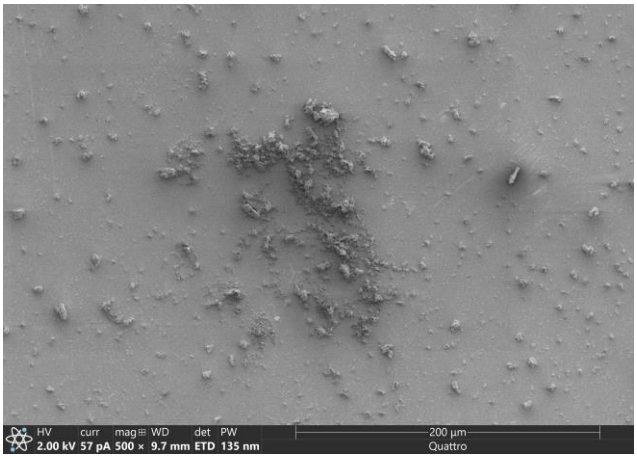
SEM Imaging

thorough examination of the study samples was conducted using a Scanning Electron Microscope (SEM) to analyze their surface structure at the microscopic level. This process enabled us to obtain high-resolution images that revealed the fine morphological features of the surface, including structural distribution, grain size, and porosity. These findings significantly deepened our understanding of the physical and chemical properties of the samples and guided our analysis toward a more accurate interpretation of their behavior in proposed practical applications.

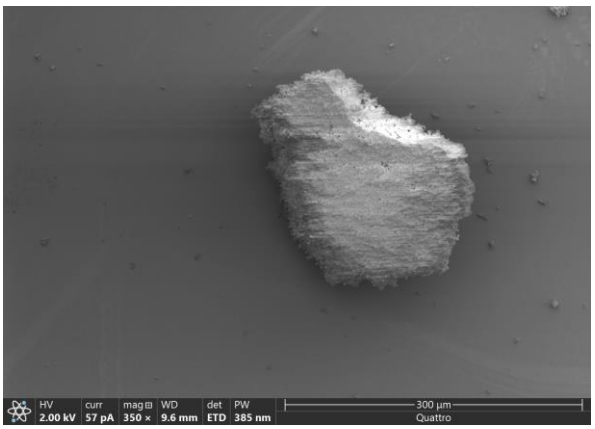




Co@g-C₃N



Cu@g-C₃N



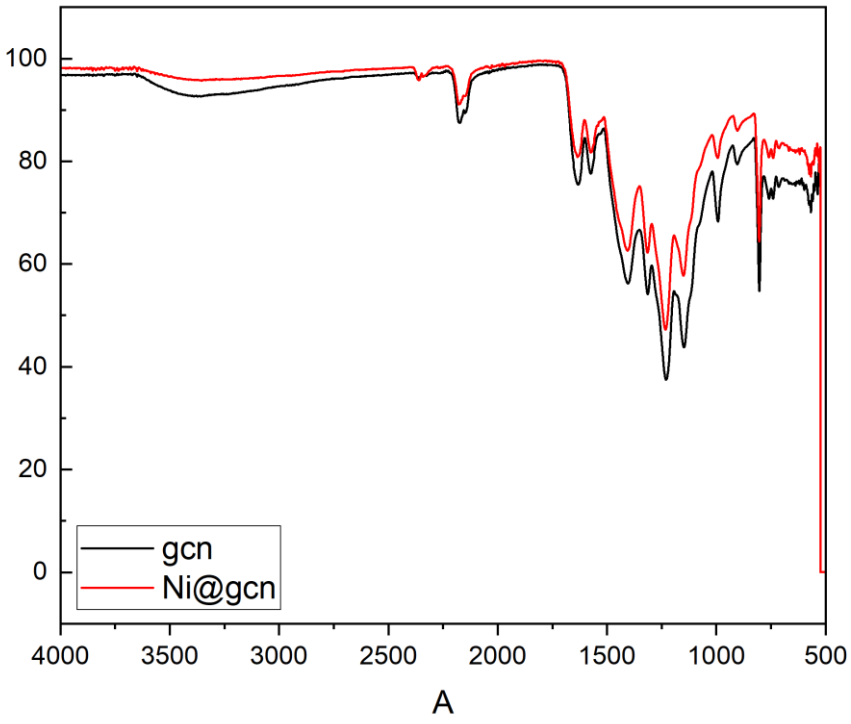
جامعة الملك عبدالله
للعلوم والتقنية
King Abdullah University of
Science and Technology

27th – 31st July

This week, I began characterizing the samples obtained from the LnNi-PHI catalyst experiment. We used the FTIR device, through which I learned more about how it operates. FTIR is used to analyze chemical compounds by measuring infrared absorption, which helps identify functional groups present in the sample and understand the molecular structure of the material.



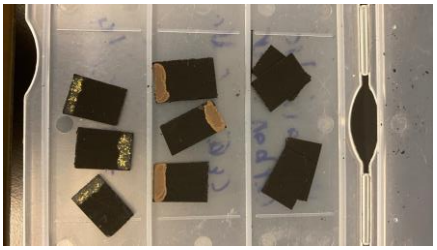




This week, a fresh batch of the Ni@g-CN catalyst was prepared and cooked. A CuCl-based catalyst was also prepared by mixing 2 grams of melamine with 100 mg of CuCl.

❖ Ink Preparation and Electrode Coating

Prepared catalyst ink by mixing:
– 0.003 g of catalyst powder
– 1 mL ethanol
– 1 mL distilled water
– 15 µL Nafion
Applied the ink onto a carbon paper strip measuring 2 cm in length. The ink was drop only on the tip (0.5 cm area).
Used a total loading of 167 µL, applied in five steps:
– Four drops of 20 µL each
– One final drop of 7 µL

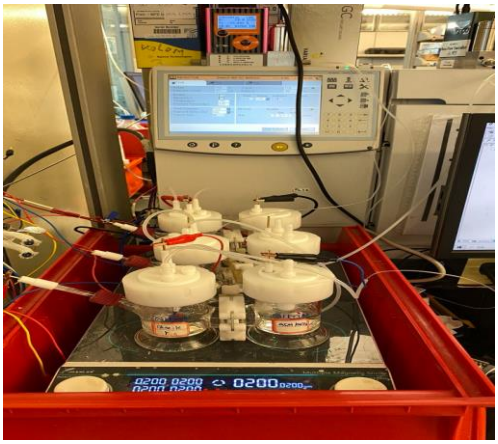


❖ Electrochemical Testing

Performed electrochemical tests using $KHCO_3$ solution. Each ctalyst was tested three times using different voltages:
-1.7 V
-1.5 V
-1.3 V

Each run followed the same procedure:

– Activation phase: 30 minutes at -1.64 (stable across all runs)
– Pause the test (waits)
– Collect a sample from the solution for NMR
– Replace the solution and prepare the cell
– Purging phase: 10 minutes
– Main test run: 1 hour
– Start GC 10 minutes after the run begins



Sample Preparation for NMR

After each electrochemical test, samples were prepared for NMR by mixing:

– 500 µL of the collected solution
– 10 µL DMSO
– 50 µL D_2O

The mixture was transferred into **NMR tubes** for analysis.



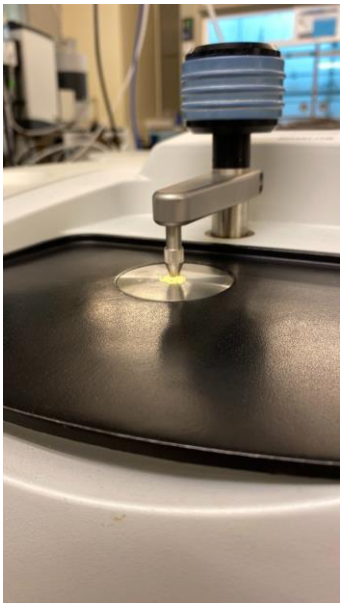


Material Characterization

Performed **FTIR** analysis to confirm functional group presence in the catalyst.
Conducted **XRD** to examine the crystal structure of the newly synthesized catalyst materials.







Photocatalytic Test of LnNi-PHI

Sample Preparation

20 mg of C_3N_4 was mixed with 3 mL of methanol and 27 mL of distilled water. The mixture was sonicated for a few minutes in an ultrasonic bath to ensure uniform dispersion and a stable suspension

Light Source and Setup

The experiment used a Lamp, which emits high-intensity white light simulating solar radiation across a broad spectrum (UV–Vis–NIR). A temperature control unit was used to maintain stable thermal conditions throughout the test.

The prepared suspension was placed in a custom photoelectrochemical cell . Before illumination, the cell was purged with argon gas to remove dissolved oxygen and create an inert atmosphere.

The cell was also connected to a GC device, a vacuum line, and gas inlet/outlet tubing for sample handling and system control. The experiment was operated continuously for 4 hours under stable illumination and temperature conditions.







.Lab Report : Procedure Modification for Improved Control of Copper Content Cu-@gcn

Following modifications to the synthesis procedure, a dry mixing approach was adopted during the initial preparation stage. This adjustment aimed to enhance control over the amount of copper introduced into the final material and to prevent excessive metal loading.

The thermal treatment was carried out using a tube furnace under the specified conditions. Upon completion of this step, solvent washing was performed to remove any residuals and ensure the purity of the final product.

Modified Synthesis Procedure:

1. Dry Mixing:

- **2 g of melamine** and **200 mg of $CuCl_2$** were mixed thoroughly without any solvents.
- The mixture was ground manually (or mechanically) until a uniform powder was obtained.

2. Thermal Treatment:

- The homogeneous powder was transferred to a **tube furnace**.
- It was heated to **550°C** at a rate of **8°C/min** under a **5% H_2 in Ar** atmosphere.
- The temperature was maintained for **4 hours**, followed by natural cooling under inert conditions.

3. Washing and Purification (Post-Furnace):

- After thermal treatment, the resulting solid was washed **twice with deionized water** to remove soluble salts.
- Then washed **twice with ethanol** to eliminate organic residues.
- **Centrifugation** was used after each washing step to separate the solid product.

4. Drying and Final Processing:

- The washed material was dried in a vacuum oven at **60°C** for **24 hours**.
- Finally, the dried product was ground into a fine powder for further characterization or application.

Electrode Preparation and Electrochemical Testing Protocol

1. Ink Deposition and Electrode Drying:

- The synthesized catalyst powder was used to prepare an ink, which was drop-cast onto **carbon paper** using **167 µL**.
- The electrode was left to dry completely to ensure uniform coating and solvent evaporation.

2. Electrochemical Activation and Sample Collection:

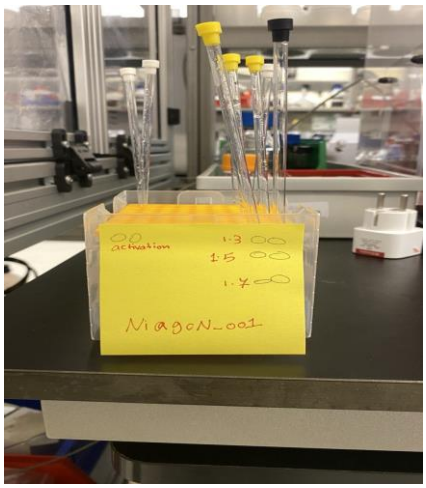
- The electrode underwent a **single 30-minute activation run** to stabilize its surface.
- After activation, a **sample was collected** for preliminary analysis.
- The electrolyte solution was then **replaced** to ensure a fresh environment for the main test

3. Purging and Main Electrochemical Test:

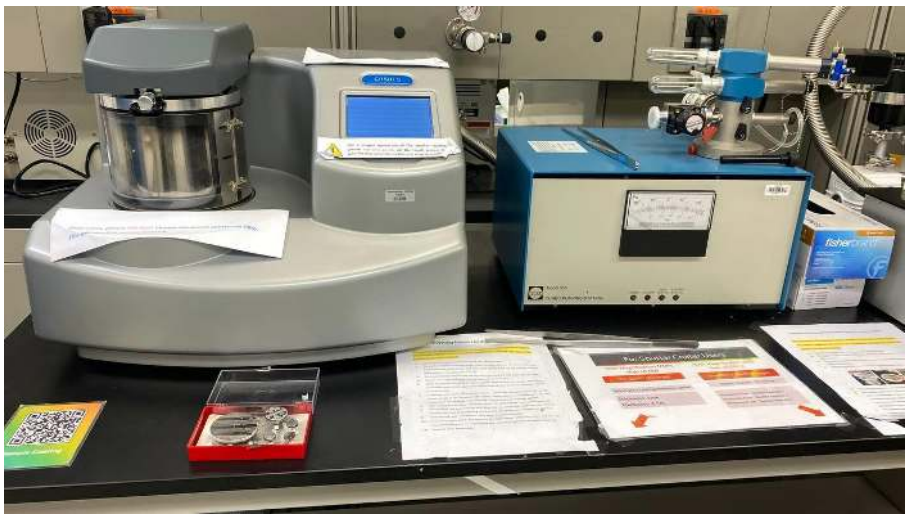
A **10-minute purge** was performed to remove any residual gases or contaminants.

- The **main electrochemical test** was initiated and conducted for **1 hour**.
- After **10 minutes** from the start of the test, **GC (gas chromatography)** was activated to begin monitoring gas evolution and product formation.

Following sample collection, the specimens were prepared for **NMR** analysis.



For **SEM characterization**, the samples were first prepared by dissolving the powdered material in ethanol to ensure complete dispersion. A small volume of the solution was then deposited onto silicon slides using a micropipette, and the slides were mounted on metal stubs suitable for SEM analysis. Prior to imaging, the samples underwent sputter coating using iridium to enhance surface conductivity. This was performed with the Q150T Sputter Coater, where the chamber was evacuated, argon gas introduced, and a thin iridium layer deposited under controlled conditions. The coated samples were then loaded into the SEM chamber for high-resolution imaging.



SEM Imaging

CN5 ,Cu@gcn-004

