

UNIVERSITY OF BAHRAIN

College of Science



Department of Chemistry

Designing Cost-effective Binary and Ternary Nanocomposites: Applications as Catalytic Materials and Humidity Sensors

A Thesis Submitted in Partial Fulfilment of the Requirements for the
M.Sc. Degree in Environmental Chemistry

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May 2024

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Publications

Shanaah, H. H., Allangawi, A., Usman, M., Idrisov, E., Ali, N., Attique, S., ... & Iqbal, J. (2024). Cobalt-Doped Graphitic Carbon Nitride: A Multifunctional Material for Humidity Sensing, Electrochemical Water Splitting and Environmental Remediation Applications. *Journal of Alloys and Compounds*, 176498.

Abstract

Graphitic carbon nitride (g-C₃N₄) emerges as a promising eco-friendly material for catalysis and sensing applications due to its unique properties. However, limitations like low specific surface area, insufficient light absorption, and poor conductivity hinder their broader usability. Elemental doping is established as an effective approach to modify the electronic structure and bandgap of g-C₃N₄ thus significantly expanding its light-responsive range for enhanced charge separation. This research involves the synthesis and validation of several materials and composites, employing characterization techniques to confirm the success of the synthesis methods. The selection of suitable materials for applications in humidity sensing and water splitting reaction prioritizes facile synthesis, good electrical conductivity, efficiency, water sensitivity, and chemical stability. The work reports on the fabrication of cost-effective and stable g-C₃N₄, cobalt-doped g-C₃N₄ (Co@g-C₃N₄), tungsten oxide embedded g-C₃N₄ (WO₃@g-C₃N₄), and silver-doped WO₃@g-C₃N₄ (Ag/WO₃@g-C₃N₄) via two simple synthesis routes: one-step calcination process, and hydrothermal treatment. The humidity sensing performance of the four sensors (g-C₃N₄, Co@g-C₃N₄, WO₃@g-C₃N₄, Ag/WO₃@g-C₃N₄) is evaluated across a broad relative humidity range (7% - 94% RH) at various testing frequencies. The doped sensors demonstrate superior humidity sensing and good reversibility compared to pristine g-C₃N₄. The Ag/WO₃@g-C₃N₄ outstand the other sensors in the humidity sensing performance. Furthermore, the g-C₃N₄ and Co@g-C₃N₄ are assessed for their suitability as electrochemical water splitting catalysts for hydrogen production, representing a step towards energy-efficient fuel cells. Notably, Co@g-C₃N₄ display enhanced performance with a lower onset potential (420 mV) and a lower Tafel slope (65.4 mV dec⁻¹) for the hydrogen evolution reaction compared to undoped counterparts. Additionally, Co@g-C₃N₄ nanorods exhibit remarkable performance for the oxygen evolution reaction, showcasing a lower onset potential (1.505 V) and a considerably low overpotential (270 mV), surpassing numerous reported electrocatalysts and even rivaling precious-metal-based ones.

Dedication

Dedicated to my parents, for their continuous love and support. For my siblings, for always cheering me up. For my friends, for always believing in me. For my husband, without whose support this accomplishment would not have been possible. Lastly, to myself, for committing to put in the effort.

Acknowledgments

In the name of Allah, the Most Gracious and the Most Merciful. Praise to be to Allah for giving me the insight and strength to complete this research. I would like to express my sincere appreciation to Dr. Javed Iqbal, for his continuous guidance, support, and welcoming manner. It has been both an honor and a privilege to conduct this research under his exceptional supervision. My sincere thanks also go to Dr. Adnan Younis, for his invaluable assistance throughout this project, and for imparting the fundamentals of proper research and writing. I am deeply grateful for the knowledge he has shared with me. Special thanks to Aluminum Bahrain B.S.C. (ALBA) for their cooperation in the analysis of our sample with the Scanning Electron Microscope (SEM). Special appreciation is extended to the University of New South Wales, in Sydney, Australia, for their collaboration with our research group, with the humidity sensing performance, and the water splitting reaction experiments. I would like to thank the head of technicians, Mr. Ali Moosa, and the technicians, Ms. Alia Mustafa, Mr. Hussain Isa, Mr. Hussain Ali, and Mr. Jassim Mohammed, for their help and assistance throughout my research journey. Lastly, my heartfelt thanks to my wonderful friends and family members who have always believed in and been proud of me. I am grateful and blessed.

List of Content

Defense Committee’s Approval.....	B
Abstract.....	D
Dedication	E
Acknowledgments	F
List of Content.....	G
List of Tables	J
List of Figures.....	K
List of Abbreviation.....	M
List of Terminologies	P
1. Chapter One: Introduction to the Study	2
1.1 Introduction.....	2
1.2 Research Problem	4
1.3 Research Questions.....	5
1.4 Research Aim and Objectives.....	6
1.5 Research Significance.....	6
1.6 The Study’s Setting	7
2. Chapter Two: Literature Review	9
2.1 The g-C ₃ N ₄ -based Humidity Sensors	9
2.2 The g-C ₃ N ₄ -based Catalysts for the Water Splitting Reaction.....	15
2.3 The Outcomes of the Literature Review	21
3. Chapter Three: Methodology and Methods	23
3.1 Methodology.....	23

3.2 Methods	24
3.2.1 Synthesis of g-C ₃ N ₄ Nanosheets	24
3.2.2 Synthesis of WO ₃ Nanoparticles	26
3.2.3 Synthesis of WO ₃ @g-C ₃ N ₄ Nanocomposite.....	28
3.2.4 Synthesis of Ag/WO ₃ @g-C ₃ N ₄ Nanocomposite.....	29
3.2.5 Synthesis of Co@g-C ₃ N ₄ Nanorods.....	31
3.2.6 Synthesis of GO Nanosheets.....	32
3.3 Instrumentation	34
3.4 Application	34
3.4.1 Assembly and measurement of humidity sensor	34
3.4.2 Measurements for the HER/OER.....	35
3.5 Statistical Methods.....	36
4. Chapter Four: Findings and Discussion	38
4.1 Material Characterization	38
4.1.1 Characterization of g-C ₃ N ₄	39
4.1.2 Characterization of WO ₃	41
4.1.3 Characterization of WO ₃ @g-C ₃ N ₄	42
4.1.4 Characterization of Ag/WO ₃ @g-C ₃ N ₄	44
4.1.5 Characterization of Co@g-C ₃ N ₄	45
4.2 Selection of the Material.....	48
4.3 Application	49
4.3.1 Humidity Sensing Performance of Selected Materials....	49
4.3.2 Water Splitting Reaction	55
4.3.2.1 HER Performance of Selected Materials.....	55

4.3.2.2 OER performance of selected materials	59
4.4 Conclusion and Key Findings.....	62
4.5 Recommendations and Future Work	63
References	66
المخلص.....	84

List of Tables

Table 1. Comparison between the Co@g-C ₃ N ₄ nanorods with g-C ₃ N ₄ based materials, and precious-metals based materials reported in literature for the HER.....	63
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List of Figures

Figure 1. The g-C ₃ N ₄ (a) before and (b) after exfoliation into nanosheets.	25
Figure 2. The WO ₃ nanoparticles after calcination.	27
Figure 3. The WO ₃ @g-C ₃ N ₄ nanocomposite after calcination.	28
Figure 4. The Ag/WO ₃ @g-C ₃ N ₄ nanocomposite after drying.	30
Figure 5. The Co@g-C ₃ N ₄ nanorods after drying.	32
Figure 6. The GO nanosheets (a) before and (b) after drying.	33
Figure 7. Schematic of the humidity sensing measurements.	35
Figure 8. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for pristine g-C ₃ N ₄	40
Figure 9. The XRD pattern for bare WO ₃ nanoparticles.	41
Figure 10. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for WO ₃ @g-C ₃ N ₄	43
Figure 11. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for Ag/WO ₃ @g-C ₃ N ₄	45
Figure 12. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for Co@g-C ₃ N ₄	47
Figure 13. The measured impedance of the four sensors (a) g-C ₃ N ₄ , (b) Co@g-C ₃ N ₄ , (c) WO ₃ @g-C ₃ N ₄ , and (d) Ag/WO ₃ @g-C ₃ N ₄ , upon exposure to various RH levels.	Error! Bookmark not defined.
Figure 14. The hysteresis of (a) g-C ₃ N ₄ , Co@g-C ₃ N ₄ , WO ₃ @g-C ₃ N ₄ , and Ag/WO ₃ @g-C ₃ N ₄ and (b) and enlarged scale for WO ₃ @g-C ₃ N ₄ , and Ag/WO ₃ @g-C ₃ N ₄	54
Figure 15. (a) HER polarization curve and (b) Tafel slope for the three electrocatalysts g-C ₃ N ₄ , Co@g-C ₃ N ₄ , and Pt.	57

Figure 16. The Volmer-Heyrovsky mechanism followed by Co@g-C ₃ N ₄ for the HER.....	58
Figure 17. Anodic polarization curves for bare g-C ₃ N ₄ and Co@g-C ₃ N ₄	60

List of Abbreviation

g-C₃N₄	graphitic carbon nitride
Co@g-C₃N₄	cobalt-doped g-C ₃ N ₄
WO₃@g-C₃N₄	tungsten oxide embedded g-C ₃ N ₄
Ag/WO₃@g-C₃N₄	silver-doped WO ₃ @g-C ₃ N ₄
RH	relative humidity
HER	hydrogen evolution reaction
OER	oxygen evolution reaction
SEM	Scanning Electron Microscope
mV/dec	millivolts per decade
C₃N₃	triazine
C₆N₇	heptazine
XRD	X-ray diffraction
TEM	Transmission Electron microscopy
FTIR	Fourier-transform infrared spectroscopy
Sec	seconds
Ag@g-C₃N₄	silver doped g-C ₃ N ₄
KIT-6	mesoporous silica solution
AgNO₃	silver nitrate
In/SnO₂@g-C₃N₄	Indium tin oxide loaded g-C ₃ N ₄
g-C₃N₄/PEO	g-C ₃ N ₄ /polyethylene oxide hybrid film
g-C₃N₄/ZnO	hybrid composite of g-C ₃ N ₄ and zinc oxide
Li@g-C₃N₄	lithium-doped g-C ₃ N ₄ composite
g-C₃N₄/GQDs	incorporation of g-C ₃ N ₄ with graphene quantum dots
BET	Brunauer-Emmett-Teller
NiCo₂O₄@g-C₃N₄	nickel cobalt oxide doped g-C ₃ N ₄

CuO/FeO@g-C₃N₄	co-doped copper oxide and iron oxide g-C ₃ N ₄
TiO₂@g-C₃N₄	titanium dioxide embedded g-C ₃ N ₄ composite
TiO₂	titanium dioxide
Ni@g-C₃N₄	nickel doped g-C ₃ N ₄ composite
EDX	Energy Dispersive X-ray spectrometry
Ir@g-C₃N₄/NG	iridium doped g-C ₃ N ₄ coupled with nitrogen-doped graphene
g-C₃N₄/MoN	g-C ₃ N ₄ and molybdenum nitride hybrid
g-C₃N₄/Cu₃P@Gr	3D-graphene matrix with g-C ₃ N ₄ and copper phosphide nanocomposite
hrs	hours
Co₃O₄@P-g-C₃N₄	cobalt oxide supported phosphorus-doped g-C ₃ N ₄ composite
((NH₄)₂HPO₄)	diammonium hydrogen phosphate
Ni(OH)₂/Ni@g-C₃N₄	nickel hydroxide and metallic nickel co-doped g-C ₃ N ₄
SnO₂@g-C₃N₄@SnS₂	tin oxide, g-C ₃ N ₄ and tin disulfide nanoshell
ZIF-67	zeolitic imidazolate framework
g-C₃N₄@ZIF-67	ZIF-67 and g-C ₃ N ₄ composite
Co₃O₄@g-C₃N₄	cobalt oxide doped g-C ₃ N ₄ composite
MOF	metal-organic framework
α-Fe₂O₃@g-C₃N₄	iron oxide embedded g-C ₃ N ₄
WO₃	tungsten oxide
GO	graphene oxide
HNO₃	nitric acid
Na₂WO₃	sodium tungstate
NaCl	sodium chloride
HCl	hydrochloric acid
EtOH	ethanol

AgCl	silver chloride
Co(NO₃)₂·6H₂O	cobalt (II) nitrate hexahydrate
NaOH	sodium hydroxide
UV	ultraviolet light
H₂SO₄	sulfuric acid
H₃PO₄	phosphoric acid
KMnO₄	potassium permanganate
H₂O₂	hydrogen peroxide
Teflon	polytetrafluoroethylene
KBr	potassium bromide
Pt	platinum
LSV	Linear sweep voltammetry
SCE	saturated calomel electrode
C	carbon
N	nitrogen
W	tungsten
O	oxygen
J	current density
η₁₀	overpotential

List of Terminologies

Humidity	The amount of water vapor present in the air (Dyer, 2012).
Composite	Two or more distinct materials present together, resulting in a new material with different properties than the original materials (Balasubramanian, 2016).
Precursor	A substance that is used initially in the synthesis or the production of another compound (Barry, Teplyakov, & Zaera, 2018).
Nanomaterial	A material that has at least one dimension within the nanoscale range of 1 – 100 nm (Kreyling, Semmler-Behnke, & Chaudhry, 2010).
Electrocatalysis	Utilizing a catalyst to accelerate an electrochemical reaction at an electrode surface (Appleby, 1983).
Electrocatalyst	The substance that accelerates an electrochemical reaction, without a permanent chemical change to the substance (Appleby, 1983).
Impedance	A measure of the opposition in a system to the flow of an alternating current at a given frequency (Lasia & Lasia, 2014).
Sensor	A device that detects changes in its surrounding environment, and sends the information to an electric device or a computer processor (Baranwal, Barse, Gatto, Broncova, & Kumar, 2022).
Hydrothermal Method	A process to fabricate a material in water under high pressure and temperature (Baig, Kammakakam, & Falath, 2021).
Autoclave	A sealed vessel or a device used in hydrothermal method to facilitate the synthesis of materials under high pressure and temperature (Baig et al., 2021).
Calcination	The process of heating solids to a high temperature, in the presence of air (EUBANK, 1951).

Tafel Slope	A plot that quantifies the relationship between the logarithm of the current density and the overpotential in an electrochemical reaction. It is commonly expressed in millivolts per decade (mV/dec) (van der Heijden, Park, Vos, Eggebeen, & Koper, 2024).
Sonication	A process that uses sound waves to produce cavities (micro-sized bubbles or voids) in a liquid due to pressure fluctuations. The cavities continuously form and collapse, and the mechanical motion helps in mixing substances efficiently (Ciesielski & Samorì, 2014).
LCR meter	An electronic equipment that is capable of measuring inductance, capacitance, and resistance of an electronic component (Callegaro, 2012).
Electrode	A conductor used in an electrochemical cell to transfer electrons from or to the electrolyte or another phase within the cell. It facilitates the loss or gain of electrons in the cell during the reaction (Bagotsky, 2005).
Linear Sweep Voltammetry	A voltammetry technique where the potential between a working electrode and a reference electrode is linearly swept in time. This technique is used to measure the current response of an electrochemical system, recording the oxidation or reduction processes of species at the electrode surface. The applied potential is incremented linearly over time from an initial potential to a final potential, and the resulting current is measured (Parker, 1986).

Chapter One: Introduction to the Study

1. Chapter One: Introduction to the Study

This chapter includes a brief introduction for the thesis. It presents the questions that are addressed during the study, and it outlines the study's primary aims and objectives. The final section of the chapter highlights the importance and significance of this research.

1.1 Introduction

Two-dimensional materials are promising candidates for various applications including catalysis and energy storage applications (Liu et al., 2021; H. Wang et al., 2023; H. Zhang et al., 2023). Among them, graphitic carbon nitride (g-C₃N₄) has received significant interest in recent years as an inexpensive, non-toxic, and environmental friendly polymeric semiconductor (Raaja Rajeshwari, Kokilavani, & Sudheer Khan, 2022). The g-C₃N₄ has 2D stacked structure as an analogue to graphite with highly delocalized π -conjugated planar structure consisting of the two basic units triazine (C₃N₃) and heptazine (C₆N₇) as shown in **Figure 1**. The units are connected by covalent bonds between carbon and nitrogen atoms (J. Wang, Hao, Ye, & Umezawa, 2017). Its high physicochemical stability, unique electronic structure, and absorptivity of visible light has made this material attractive for a variety of applications such as energy storage (Iqbal et al., 2023; Mohammad, Chandra, Khan, Choi, & Yoon, 2023; Ullah et al., 2023), sensing (Govindaraju et al., 2021; Nihal et al., 2023; Sekar, Yun, & Lee, 2023), catalysis (Kong, Wang, Ma, Sun, & Quan, 2019; Lin et al., 2023; Muhmood et al., 2024), and environmental remediation (Koutsouroubi et al., 2021; D. Li et al., 2022; Zhao, Zou, Hu, Long, & Jiao, 2021).

Within electrochemical applications, humidity sensing is one of the most extensively utilized (M. Han, Shen, & Corriou, 2024). A critical parameter in humidity measurements is relative humidity (RH), which quantifies the amount of water vapor present in air. It is expressed as the ratio between the partial pressure of water vapor and the saturated vapor pressure at a specific temperature (Blank, Eksperiandova, & Belikov, 2016). Accurate measurement and control of humidity are essential across various domains including industrial processes, agricultural practices, corrosion prevention in devices, and ensuring human comfort and health (Zhou et al., 2021). Consequently, there is a strong demand for high-performance humidity sensors that possess high sensitivity, good linearity, fast response and high selectivity (Tomer & Duhan, 2015).

Water splitting electrolysis is another crucial electrochemical process for the production of a clean energy source (Allangawi, Shanaah, Mahmood, & Ayub, 2023). Precious metal-based catalysts are the best-known electrocatalysts for the water splitting reaction as they reduce the activation energy significantly and are highly stable (Y. Gong et al., 2022). However, precious metals are not abundant on Earth and are quite costly (Yin, Rong, & Xie, 2023). Consequently, numerous attempts have been made to replace precious metal-based catalysts, and the investigation of alternative efficient catalysts is in progress (Gothandapani et al., 2023; Joshi, Pataniya, Bhadu, & Sumesh, 2023; Nangan et al., 2023; Song, Zhao, Jing, Wang, & Li, 2024).

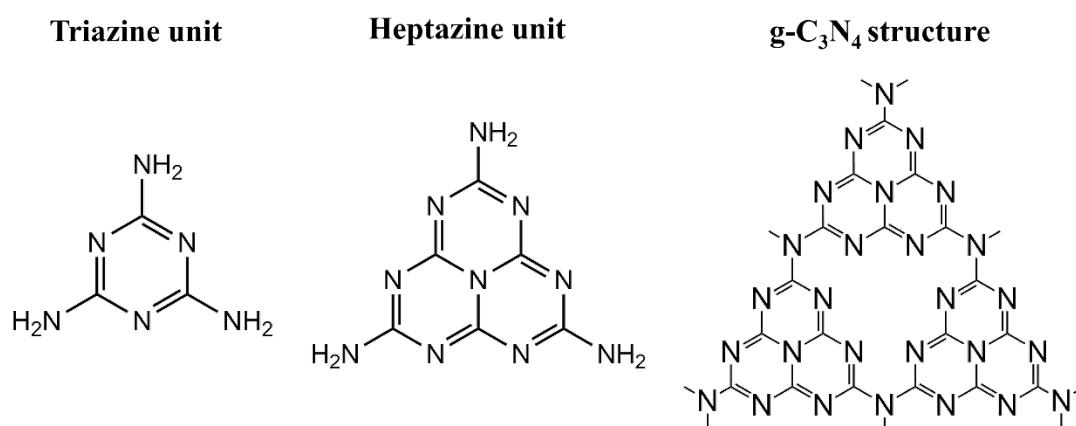


Figure 1. The structure of triazine, heptazine, and g-C₃N₄.

1.2 Research Problem

In spite of the g-C₃N₄ wide applicability, it encounters some limitations that hinder the suitability of the material in electrochemical applications. The g-C₃N₄ suffers from low specific surface area, chemical inertness, poor conductivity, and the fast recombination of photo-generated electrons (Khasim et al., 2022; Ranjithkumar et al., 2021; Zheng, Cao, & Wang, 2016). Being layered and 2D stack structured material, g-C₃N₄ has a potential affinity towards water molecules, and thus is a promising candidate for humidity sensing and water splitting reaction (Meng, Wu, Wang, & Zhang, 2020). As previously mentioned, this material has been restricted by its poor conductivity and low surface area (Meng et al., 2020; X. Wang et al., 2019). As such, investigating the humidity sensing capabilities of an enhanced, doped g-C₃N₄ would be of interest.

Numerous strategies were implemented to overcome the inadequacies of g-C₃N₄. Among those, doping, metal deposition, morphology modification, and heterojunction construction were found to be very effective (Al-Najar et al., 2022; Ali et al., 2024; Hussain et al., 2022; S. Wang et al., 2022; Xing et al., 2022; Younis et al., 2024; X. Zhang et al., 2023). The introduction of foreign atoms modifies electrons distribution in g-C₃N₄ structure, and promote the effective migration of electrons, thus achieving the purpose of inhibiting the recombination of photogenerated electrons and holes (Chen et al., 2024; Pham, Park, & Kim, 2021). The surface area of the g-C₃N₄ can also be improved tremendously by the exfoliation of g-C₃N₄ or morphology modifications (Cai et al., 2020; Joy, George, Poornima Vijayan, Anas, & Thomas, 2023; K. Li et al., 2017). Overall, it is highly recommended to modify the g-C₃N₄ before conducting electrochemical studies.

1.3 Research Questions

This thesis pursues to answer how the synthesis of g-C₃N₄ and its composites can enhance their electrical conductivity and photocatalytic efficiency for applications in humidity sensing and water splitting. It seeks to examine how the elemental doping impacts the properties of g-C₃N₄-based composites in terms of their structural, chemical, and functional characteristics, as well as to what extent the synthesized nanomaterials improve the efficiency of the electrochemical studies. Lastly, it seeks to answer which of the synthesized materials demonstrates the most effective humidity sensing performance and water splitting catalysis.

1.4 Research Aim and Objectives

In this work, we report the synthesis of low-cost g-C₃N₄ via a simple one-step calcination method. The surface area and active sites of g-C₃N₄ were increased by exfoliation with an acid. Metal doping of g-C₃N₄ was carried out via a green hydrothermal method. The doping opted to increase overall conductivity which in turn modified the morphology of the g-C₃N₄. Thus, improved sensing response and catalytic activities were recorded.

1.5 Research Significance

This thesis seeks to contribute to the field of material science by developing an understanding of how elemental doping and synthesis methods affect the properties of g-C₃N₄-based nanomaterials. Through enhancing the materials' efficiency in the water splitting reaction and humidity sensing, this study contributes to environmental monitoring and sustainable energy generation. The development of water splitting catalysts and more dependable humidity sensors has the potential to improve the accuracy of environmental sensing and increase the efficiency of hydrogen fuel production. Particularly, the study addresses the current gap in research regarding the practical application of g-C₃N₄ and its composites, particularly in the context of energy and environmental monitoring.

1.6 The Study's Setting

The synthesis and the characterization of the synthesized materials has taken place in the College of Science, University of Bahrain. The Scanning Electron Microscope (SEM) analysis was performed at Aluminum Bahrain B.S.C. (ALBA). Additionally, a collaborative effort between the University of Bahrain and the University of New South Wales, in Sydney, Australia, facilitated the experiments related to humidity sensing and water splitting experiments.

Chapter Two: Literature Review

2. Chapter Two: Literature Review

This chapter consists of three sections that will discuss the most important attempts in the construction of g-C₃N₄-based humidity sensors and catalysts for the water splitting reaction, the hydrogen evolution reaction (HER) and the oxygen evolution reaction (OER). The chapter will conclude with the outcomes of the literature review, and the benefits acquired from the previously conducted research.

2.1 The g-C₃N₄-based Humidity Sensors

Humidity detection is crucial for a wide range of applications across various fields, including industrial processes, agriculture, healthcare, and environmental monitoring. Precise humidity measurement ensures optimal operation conditions and product quality in industrial settings, helps maintain appropriate conditions for crop growth in agriculture, prevents corrosion and degradation of materials, and is vital for human comfort and health (Arman Kuzubasoglu, 2022; Ma, Fei, & Zhang, 2023). Common examples of humidity sensors include metal oxides (Shaheen et al., 2020), polymers (Fratoddi, Bearzotti, Venditti, Cametti, & Russo, 2016), and carbon-based materials (Tulliani, Inserra, & Ziegler, 2019). Therefore, we are interested in exploring the use of g-C₃N₄-based composite materials as humidity sensors.

A study published in 2014 by Zhang Z. et al, tested the humidity sensing performance of intercalated g-C₃N₄ nanosheets (Z. Zhang, Huang, Yuan, & Dong, 2014). The synthesis of g-C₃N₄ was accomplished by the thermal polymerization of urea in the presence of LiCl as an intercalated agent at 550°C. The intercalated g-C₃N₄ nanosheets exhibited good humidity sensitivity, with a response time of 0.9 seconds (sec). The study emphasized the potential of g-C₃N₄-based composites to become effective humidity sensors.

An article published in 2016 by Tomer K. et al, developed a cubic mesoporous silver doped g-C₃N₄ (Ag@g-C₃N₄) to Ag@g-C₃N₄ monitor humidity (Tomer, Thangaraj, Gahlot, & Kailasam, 2016). The mesoporous g-C₃N₄ was obtained by the calcination of cyanamide and mesoporous silica solution (KIT-6) at 550°C. Silver nanoparticles were incorporated into the mesoporous g-C₃N₄ by soaking the g-C₃N₄ in a silver nitrate (AgNO₃) solution, followed by the reduction of Ag ions to Ag nanoparticles. Moreover, the Ag@g-C₃N₄ exhibited high sensitivity over the range of (11%-98% RH) with a 3 sec response time. The sensor retained high stability and low hysteresis.

Following the attempt made by Tomer K. et al in 2016 (Tomer et al., 2016), Malik R. et al. published an article presenting a cubic mesoporous Indium tin oxide loaded g-C₃N₄ (In/SnO₂@g-C₃N₄) humidity sensor (Malik et al., 2017). The composite was synthesized in a one-step calcination method. A tin salt, indium salt, and cyanamide were impregnated into the KIT-6 template as precursors for g-C₃N₄, SnO₂, and In, respectively. The impregnated template was calcinated at 550°C to yield In/SnO₂@g-C₃N₄. The ordered In/SnO₂@g-C₃N₄ structure was characterized by X-ray diffraction (XRD), and nitrogen adsorption-desorption isotherms to confirm the mesoporous structure. The In/SnO₂@g-C₃N₄ sensor exhibited excellent sensitivity over a range of 11%-98% RH, and demonstrated a rapid response time of 3.5 sec.

A study conducted in 2021 by Zhou L. et al, designed a humidity sensor based on g-C₃N₄/polyethylene oxide (g-C₃N₄/PEO) hybrid film (Zhou et al., 2021). The g-C₃N₄ was obtained by the simple calcination of urea at 550°C. On the other hand, the g-C₃N₄/PEO hybrid film was completed by spin coating a mixture of g-C₃N₄ and PEO solution. The g-C₃N₄/PEO film showed a broad humidity detection range (11%-97% RH), and a rapid response of 2.2 sec at 97% RH.

A research published in 2021 by Yu S. et al. (S. Yu, Chen, Zhang, Zhang, & Liu, 2021), fabricated a hybrid composite of g-C₃N₄ and zinc oxide (g-C₃N₄/ZnO) specifically designed for detecting human breath. The g-C₃N₄/ZnO composite was synthesized using the chemical vapor deposition technique. The study's main results showed that the sensor displayed high sensitivity, minimal hysteresis, and quick response time across a wide range of humidity levels (11%-95% RH), making it an effective tool for diagnosing respiratory conditions.

A study in 2022 by Tseng S. and Tsai Y. (Tseng & Tsai, 2022), aimed to develop a metal-free, sensitive humidity sensor using lithium-doped g-C₃N₄ composite (Li@g-C₃N₄) on a porous graphene electrode. The synthesis of g-C₃N₄ involved a one-step calcination of urea at 500°C, while Li@g-C₃N₄ was synthesized using a lithium salt precursor in a hydrothermal treatment. The humidity sensing performance of the Li@g-C₃N₄ across a wide range of relative humidity levels (11-97% RH), demonstrated a rapid response, high sensitivity, and minimal hysteresis of the fabricated sensor. The sensor was also successfully applied in human breath detection, non-contact sensing of a fingerprint, and skin humidity detection.

Morsy M. et al. published research in 2023 on the incorporation of g-C₃N₄ with graphene quantum dots (g-C₃N₄/GQDs) for humidity sensing (Morsy, Gomaa, Mokhtar, ElHaes, & Ibrahim, 2023). The synthesis of the g-C₃N₄ involved a one-step calcination of urea at 550°C, whereas GQDs were prepared hydrothermally using citric acid as a carbon source. The synthesis of g-C₃N₄/GQDs was accomplished by ultrasonication. The composite exhibited improved humidity sensing performance compared to individual components. The sensor demonstrated good reversibility and fast response time across a wide range of relative humidity (7%-97% RH), making it highly suitable for practical applications such as humidity alarm devices and breath analysis.

A study by Gong L. et al. in 2023, researched the performance of nickel cobalt oxide doped g-C₃N₄ (NiCo₂O₄@g-C₃N₄) for humidity detection (L. Gong et al., 2023). The synthesis of the g-C₃N₄ was completed by the calcination of urea at 550°C. The NiCo₂O₄@g-C₃N₄ was prepared by stirring cobalt and nickel salts with g-C₃N₄, followed by calcination. The nanocomposite was then tested for its humidity sensing performance, demonstrating high sensitivity, and a fast response of 7 seconds (sec) at 67% RH.

In 2023, a study was conducted by Khasim S. et al. to develop a co-doped copper oxide and iron oxide g-C₃N₄ heterostructure (CuO/FeO@g-C₃N₄) for humidity sensing (Khasim et al., 2023). The synthesis of g-C₃N₄ was conducted by the thermal polymerization of melamine at 550°C. The CuO/FeO@g-C₃N₄ heterostructure was synthesized hydrothermally by incorporating the metal oxides. The CuO/FeO@g-C₃N₄ heterostructure exhibited sensitive humidity detection and fast response time of 26 sec at 67% RH. The multifunctional composite was also applied for the detection of methane gas at ppb levels and soil moisture content.

In a 2023 study published by Maluangnont T. et al, titanium dioxide embedded g-C₃N₄ composite (TiO₂@g-C₃N₄) was investigated for humidity detection (Maluangnont et al., 2023). The fabrication of g-C₃N₄ was achieved by simple calcination of melamine at 550°C. The incorporation of TiO₂ into g-C₃N₄ was done by solvothermal method. The humidity sensing for the composite was inspected. The TiO₂@g-C₃N₄ showed a decrease in electrical resistance with higher humidity exposure. The doping of TiO₂ has enhanced the conductivity of the pristine g-C₃N₄.

2.2 The g-C₃N₄-based Catalysts for the Water Splitting Reaction

Hydrogen energy has recently gained much interest as a green-sustainable alternative to fossil fuels (Allangawi et al., 2023). The advantages of hydrogen energy revolve around the fact that its only byproduct is water, as it is based on combining hydrogen and oxygen gases to produce water and energy. The water splitting reaction is ideally utilized to produce high purity hydrogen and oxygen gases, where it consists of two half-electrode reactions, the cathodic HER for hydrogen production and the anodic OER for oxygen production. Therefore, it is of interest to synthesize cheap catalysts for realizing the full potential of the water splitting reaction (Sultan et al., 2019).

A coral-like structured nickel doped g-C₃N₄ composite (Ni@g-C₃N₄) was developed by Wang L. et al. in 2017 (L. Wang et al., 2017). The study aimed to produce a good electrocatalyst for the HER in an alkaline medium with an easily scalable synthesis route. The synthesis of g-C₃N₄ was accomplished by the thermal polymerization of melamine at 550°C under nitrogen, followed by ball and sand milling. The Ni@g-C₃N₄ was synthesized using an electrodeposition method. Lastly, the HER activity results showed that the Ni@g-C₃N₄ has an overpotential of 222 mV at a current density of 10 mA cm⁻², and a Tafel slope of 128 mV dec⁻¹.

The electrocatalytic properties of iridium doped g-C₃N₄ coupled with nitrogen-doped graphene (Ir@g-C₃N₄/NG) for water splitting was investigated by Jiang B. et al. in 2018 (Jiang et al., 2018). The study employed a solvothermal method for the synthesis of the composite. The electrocatalytic results for the water splitting reaction by the Ir@g-C₃N₄/NG composite were excellent. For the HER, the Ir@g-C₃N₄/NG composite showed an overpotential of only 22 mV at a current density of 10 mA cm⁻², and a Tafel slope of 22 mV dec⁻¹. For the OER, the Ir@g-C₃N₄/NG composite showed an overpotential of 300 mV at the same current density, and a Tafel slope of 72.8 mV dec⁻¹. The Ir@g-C₃N₄/NG composite can be highly efficient for the HER and the OER. However, the use of rare and precious metals like iridium may hinder its application.

A study in 2018 by Jin H. et al. (Jin et al., 2018), investigated the construction of a two-dimensional g-C₃N₄ and molybdenum nitride hybrid (g-C₃N₄/MoN) for the electrocatalytic HER. The synthesis of g-C₃N₄/MoN was executed by the thermal polymerization of MoN dispersion and dicyandiamide mixture at 550°C under argon. The HER studies revealed that the g-C₃N₄/MoN hybrid demonstrated a low overpotential of 110 mV at a current density of 10 mA cm⁻², and a Tafel slope of 57.8 mV dec⁻¹. However, molybdenum is considered a rare metal, which may hinder the application of the g-C₃N₄/MoN in the HER. The Ir@g-C₃N₄/NG previously mentioned (Jiang et al., 2018) has surpassed the performance of g-C₃N₄/MoN, which may be attributed to the presence of the highly active Ir Metal.

A study in 2019 by S. Riyajuddin et al. (Riyajuddin, Tarik Aziz, Kumar, Nessim, & Ghosh, 2020) developed a noble-metal free electrocatalyst for the water splitting reaction using a 3D-graphene matrix with g-C₃N₄ and copper phosphide (g-C₃N₄/Cu₃P@Gr) nanocomposite. The synthesis of g-C₃N₄ was achieved by the thermal polymerization of dicyandiamide at 550°C, followed by exfoliation of g-C₃N₄ via sonication in isopropanol yielding g-C₃N₄ nanosheets. The HER and the OER performance of the g-C₃N₄/Cu₃P@Gr nanocomposite was recorded. The g-C₃N₄/Cu₃P@Gr electrocatalyst exhibited an overpotential of 67 mV at a current density of 10 mA cm⁻² for the HER, and a Tafel slope of 45 mV dec⁻¹. For the OER, the electrocatalyst showed an overpotential of 255 mV at the same current density, and a Tafel slope of 40 mV dec⁻¹. The g-C₃N₄/Cu₃P@Gr electrocatalyst sustained excellent stability with minimal loss in potential after 35 hours (hrs), making it a decent candidate for practical applications.

A study in 2019 led by M. Zhu et al. (M. Zhu et al., 2019) fabricated cobalt oxide supported phosphorus-doped g-C₃N₄ composite (Co₃O₄@P-g-C₃N₄) for the electrocatalysis of the OER. The synthesis of P-g-C₃N₄ was accomplished by heating melamine and diammonium hydrogen phosphate ((NH₄)₂HPO₄) at 450°C. The Co₃O₄@P-g-C₃N₄ was obtained by a hydrothermal method using a cobalt salt as a precursor. The electrocatalysis of the Co₃O₄@P-g-C₃N₄ composite recorded a low overpotential of 320 mV at a current density of 10 mA cm⁻² for the OER, and a Tafel slope of 66.8 mV dec⁻¹. The Co₃O₄@P-g-C₃N₄ has proved to be a proper candidate for the OER as a non-precious metal electrocatalyst.

In 2021, J. Zhang et al. conducted a study on the electrocatalysis of the HER utilizing nickel hydroxide and metallic nickel co-doped g-C₃N₄ (Ni(OH)₂/Ni@g-C₃N₄) (J. Zhang, Zhao, Wu, Li, & Wang, 2021). The synthesis of g-C₃N₄ involved the calcination of urea at 520°C, followed by ultrasonication for the exfoliation of g-C₃N₄ to g-C₃N₄ nanosheets. The Ni(OH)₂ and Ni co-doping was accomplished by in-situ reduction of nickel salt, in a suspension of the previously synthesized g-C₃N₄. The characterization confirmed the successful co-doping of Ni(OH)₂ and Ni particles into the g-C₃N₄ framework. For the HER performance, the Ni(OH)₂/Ni@g-C₃N₄ composite recorded an overpotential of 1881 mV at a current density of 100 mA cm⁻², and a Tafel slope of 44.3 mV dec⁻¹. The obtained overpotential for the composite was shown to be superior to that of the bare g-C₃N₄ and the Pt catalyst. However, the reliability of the results in this study is in question, as the obtained overpotential of 1881 mV is extremely high, deeming the catalyst as inactive.

Ultrafine g-C₃N₄ quantum dots were fabricated for use as a catalyst for the HER by N. Yang et al. in 2022 (Yang, Chen, Zhao, & Cui, 2022). The novel synthesis method for the ultrafine g-C₃N₄ quantum dots involved the calcination of melamine at 550 °C. Then, the bulk g-C₃N₄ was electrochemically exfoliated using Al³⁺ as an intercalating agent, and oleic acid as a stabilizing agent. The particle size was 3.5 nm with a thickness of ~1 nm. The HER results were recorded for the g-C₃N₄ quantum dots, revealing an outstanding low overpotential 208 mV at a current density of 10 mA cm⁻², and a Tafel slope of 52 mV dec⁻¹. The results highlight the potential of g-C₃N₄ quantum dots as efficient, metal-free catalysts for the HER.

In 2022, B. Zhang et al. fabricated a hollow nanosphere of an outer shell of tin oxide, followed by g-C₃N₄ and tin disulfide as inner shells, respectively (SnO₂@g-C₃N₄@SnS₂) (B. Zhang et al., 2022). A step-by-step self-assembly method was adopted to create the SnO₂@g-C₃N₄@SnS₂ nanospheres, utilizing SiO₂ nanospheres as templates. The hollow nanospheres electrocatalysts were tested for the HER and the OER in an alkaline medium. The SnO₂@g-C₃N₄@SnS₂ revealed an overpotential of 386 mV and 475 mV at a current density of 10 mA cm⁻² for the HER and the OER, respectively. The Tafel slopes were 291 mV dec⁻¹ and 253 mV dec⁻¹ for the HER and the OER, respectively.

A zeolitic imidazolate framework (ZIF-67) and g-C₃N₄ composite (g-C₃N₄@ZIF-67) was assembled by S. Khan et al. in 2023 for the overall water splitting reaction (Khan, Noor, Iqbal, Pervaiz, & Yaqoob, 2023). The g-C₃N₄ was prepared by the calcination of melamine at 550°C under argon. The g-C₃N₄@ZIF-67 composite was synthesized by hydrothermal method involving cobalt salt and 2-methylimidazole with g-C₃N₄ as precursors. The g-C₃N₄@ZIF-67 electrocatalyst showed an overpotential of -176 mV at a current density of 10 mA cm⁻² for the HER, and a Tafel slope of 165 mV dec⁻¹. However, it is unclear why the overpotential was recorded as a negative value as it opposes the definition of the overpotential in practical condition. The overpotential is defined as the additional potential needed to drive the reaction at a practical rate compared to the thermodynamic equilibrium potential (Bard, Faulkner, & White, 2022). On the other hand, the electrocatalyst showed an overpotential of 255 mV at a current density of 10 mA cm⁻², and a Tafel slope of 90 mV dec⁻¹ for the OER.

A study in 2023 by L. Fan et al, explored the performance of cobalt oxide doped g-C₃N₄ composite (Co₃O₄@g-C₃N₄) towards the electrocatalysis of the OER (Fan et al., 2023). The g-C₃N₄ was initially synthesized by thermal polymerization of urea at 550°C under nitrogen gas. The incorporation of Co₃O₄ into g-C₃N₄ was accomplished via ultrasonication. The electrocatalytic performance of the Co₃O₄@g-C₃N₄ towards the OER revealed an overpotential of 627 mV at a current density of 10 mA cm⁻², and a Tafel slope of 54 mV dec⁻¹. Other precious-metal free composites previously mentioned (Khan et al., 2023; Riyajuddin et al., 2020; B. Zhang et al., 2022) had lower overpotentials for the OER. However, the Co₃O₄@g-C₃N₄ exhibited decent stability performance, with only 5.4% performance decay after 1000 cyclic voltammetry cycles.

A study in 2024 conducted by S. Kumaravel et al. (Kumaravel et al., 2024), developed a metal-organic framework (MOF) derived iron oxide embedded g-C₃N₄ (α -Fe₂O₃@g-C₃N₄) for the OER. The synthesis of g-C₃N₄ involved the thermal annealing of melamine at 550°C. The MOF derived α -Fe₂O₃@g-C₃N₄ was fabricated via hydrothermal treatment and calcination. The electrocatalyst performance towards the OER was recorded, and the main results revealed a low overpotential of 290 mV at a current density of 10 mA cm⁻², and a Tafel slope of 64 mV dec⁻¹. The main results suggest the potential of the MOF derived α -Fe₂O₃@g-C₃N₄ as an effective and durable electrocatalyst.

2.3 The Outcomes of the Literature Review

The literature review has concluded several important key findings:

- Notably, the studies employing exfoliated g-C₃N₄ nanosheets have demonstrated superior performance compared to bulk g-C₃N₄. Therefore, exfoliation of bulk g-C₃N₄ will be performed to increase the overall surface area and thus the effectiveness of our material.
- Additionally, the utilizing of urea as a precursor for g-C₃N₄ is identified as a promising and cost-effective approach, given its availability and affordability within the university's resources. Using urea as the main precursor will hopefully facilitate the large-scale production for g-C₃N₄.
- The established characterization techniques within the literature will be utilized in this research, to validate and confirm the synthesis methods. The characterization techniques will allow us to explore the properties of the synthesized materials.
- Most humidity sensing studies have focused on relative humidity; thus, this research will continue to employ relative humidity as a measure in its experimental studies.
- The use of hydrothermal method will be employed in the study, as it is an environmentally friendly synthesis route, cost-effective, with no requirement of utilizing organic solvents.

Chapter Three:

Methodology and Methods

3. Chapter Three: Methodology and Methods

The methodology and methods used to accomplish the objectives of the project are covered in this chapter. This chapter also describes the details of materials' synthesis, the characterization instruments, and the statistical methods. Additionally, it explains the selection criteria for the experimental protocols.

3.1 Methodology

The selection criteria for the synthesis method emphasized simplicity, cost-effectiveness, and scalability. Hydrothermal synthesis was chosen for its convenience, as it does not require sophisticated instruments and uses water rather than organic solvents, making the process more environmentally friendly (Nandagudi et al., 2022). Key parameters in hydrothermal synthesis such as temperature, reactant concentration, and reaction duration are easily controlled, facilitating scalability without substantial energy demands (Mirzaei & Neri, 2016).

Additionally, calcination was also employed in some cases to enhance the crystallinity of the materials, remove impurities, and increase porosity, thereby expanding the surface area (N. Fu, Chen, Chen, Ding, & Ren, 2023; Vasiljević et al., 2021).

The cost of the project was also a priority, utilizing inexpensive, readily available chemicals from the University of Bahrain's existing supplies to minimize project expenses.

Characterization of the synthesized materials is important for validating the methods used and ensuring the quality of the product. Widely utilized in nanomaterial research, the chosen instruments enable the analysis of morphology, chemical, and elemental composition (Carrillo-Gaytan, Rivera-Rangel, Razo-Lazcano, Falcón-Millán, & González-Muñoz, 2024; Mourdikoudis, Pallares, & Thanh, 2018; Samar, Kumar, & Kumar, 2024). The characterization was performed prior to the application of materials in humidity sensing and water splitting reactions, ensuring the materials meet the required specifications.

3.2 Methods

The following section discusses the details and stepwise protocols conducted for the synthesis of the materials.

3.2.1 Synthesis of g-C₃N₄ Nanosheets

The fabrication of g-C₃N₄ nanosheets was executed in accordance with previously established protocol (X. Zhu, Duan, Wang, Xin, & Song, 2022), with minor changes. In a crucible, a generous amount of urea was calcinated at an elevated temperature of 550°C for a duration of 5 hrs, to yield bulk g-C₃N₄ nanosheets. Subsequently, approximately 3.00 g of the resultant bulk g-C₃N₄ was refluxed in a 50 mL solution of 5 M nitric acid (HNO₃) maintained at 110°C overnight. The obtained solution was then washed multiple times with distilled water until a near neutral pH was achieved. The final product was then allowed to air-dry overnight. The exfoliated g-C₃N₄ yielded an average of 2.00 g for every 100 g of urea.. The g-C₃N₄ before and after exfoliation into nanosheets is presented in **Figure 1**.

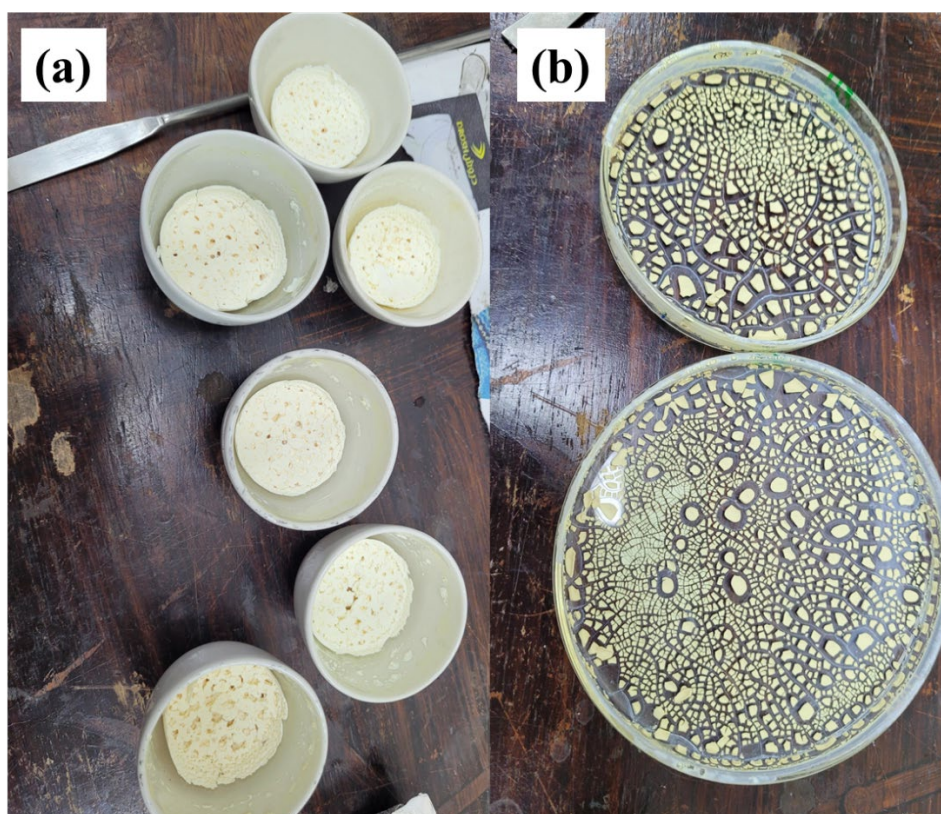


Figure 1. The g-C₃N₄ **(a)** before and **(b)** after exfoliation into nanosheets.

3.2.2 Synthesis of WO₃ Nanoparticles

The synthesis of WO₃ was performed utilizing a hydrothermal technique, following a method similarly described by (Y. Yu, Zeng, Xu, & Peng, 2016). Initially, 2.00 g of sodium tungstate (Na₂WO₃) and 0.76 g of sodium chloride (NaCl) were dissolved in 30 mL distilled water until total dissolution. Subsequently, concentrated hydrochloric acid (HCl, 37% by weight) was incrementally added under continuous stirring until a change in the solution's color was observed from a turbid white to a greenish-yellow, indicating a pH of approximately 2. The solution was transferred to a sealed autoclave and subjected to heating at 180°C for 24 hrs. Following hydrothermal treatment, the solution was centrifuged, and the precipitate was repeatedly washed with distilled water and ethanol (EtOH) to remove impurities. The washed precipitate was dried at 80°C for an overnight period. Finally, the dried product underwent calcination in a muffle furnace at 450°C for 2 hrs to yield 1.30 g of the WO₃ nanoparticles, shown in **Figure 2**.



Figure 2. The WO₃ nanoparticles after calcination.

3.2.3 Synthesis of $\text{WO}_3@\text{g-C}_3\text{N}_4$ Nanocomposite

The binary $\text{WO}_3@\text{g-C}_3\text{N}_4$ nanocomposite was accomplished in accordance with previously established method (Jilani et al., 2023). In the beginning, 0.70 g of $\text{g-C}_3\text{N}_4$, prepared in a preceding step, was uniformly dispersed in 300 mL of distilled water. To this dispersion, concentrated HCl was added dropwise under continuous magnetic stirring until the pH of the solution dropped to 2. Afterwards, 0.70 g of the previously prepared WO_3 nanoparticles were introduced to the solution. The resultant mixture was sonicated for 30 minutes (min) and centrifuged a few times with distilled water to achieve a neutral pH. The precipitate was dried overnight at 100°C . Finally, the dried product underwent calcination at 500°C for a duration of 4 hrs as shown in **Figure 3**. The average mass of a batch was 0.52 g of $\text{WO}_3@\text{g-C}_3\text{N}_4$ nanocomposite.

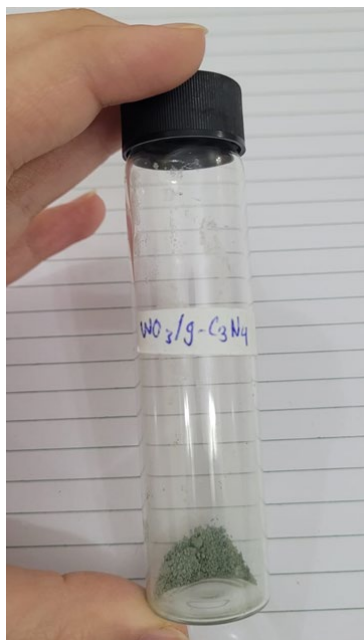


Figure 3. The $\text{WO}_3@\text{g-C}_3\text{N}_4$ nanocomposite after calcination.

3.2.4 Synthesis of Ag/WO₃@g-C₃N₄ Nanocomposite

The synthesis of the ternary Ag/WO₃@g-C₃N₄ nanocomposite, as modified from (Pu et al., 2024) protocol, employs a hydrothermal treatment rather than calcination, resulting in a product containing metallic Ag instead of silver chloride (AgCl). Initially, 20.0 mg of AgNO₃ and 150 mg of WO₃ nanoparticles were homogenously dispersed in 20 mL distilled water, followed by sonication for 30 min. Concurrently, 150 mg of g-C₃N₄ was dispersed in an identical volume of distilled water and sonicated for the same duration. Subsequently, the two solutions were combined and further sonicated for 30 min to ensure thorough mixing. The solution was transferred to an autoclave and heated at 150°C for 24 hrs. Upon completion, the product was isolated by centrifugation and washed with distilled water multiple times until neutrality was achieved. The precipitate was allowed to air-dry at room temperature overnight. The initial synthesis batch yielded 0.02 g of the Ag/WO₃@g-C₃N₄ nanocomposite. In an attempt to improve the yield, the quantities of the initial precursors were increased threefold for the second batch, which consequentially led to an enhanced yield of approximately 0.77 g. The Ag/WO₃@g-C₃N₄ nanocomposite is shown in **Figure 4**.

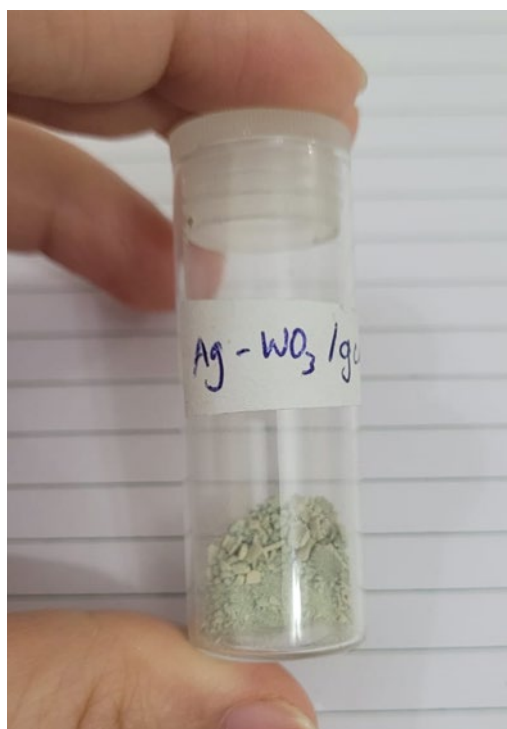


Figure 4. The Ag/WO₃@g-C₃N₄ nanocomposite after drying.

3.2.5 Synthesis of Co@g-C₃N₄ Nanorods

The synthesis of the Co@g-C₃N₄ nanorods was accomplished in accordance with previously published protocol (Shu et al., 2015), with minor changes. In separate beakers, around 1.00 g of the previously synthesized g-C₃N₄ and cobalt (II) nitrate hexahydrate (Co(NO₃)₂·6H₂O) were separately dissolved in 10 mL distilled water. Both solutions were sonicated at a controlled temperature of 35°C for 10 min. Upon mixing the two solutions, an additional volume of 20 mL of distilled water was added. The mixed solution was further sonicated under identical conditions, followed by a hydrothermal treatment in an autoclave for 16 hours at 160°C. The obtained Co@g-C₃N₄ nanorods were washed with distilled water, filtered, and dried. The procedure yielded nearly 0.80 g of Co@g-C₃N₄ nanorods as shown in **Figure 5**.



Figure 5. The Co@g-C₃N₄ nanorods after drying.

3.2.6 Synthesis of GO Nanosheets

The synthesis of GO was prepared using Hummer's method (Marcano et al., 2010). First, 7.00 g of pure graphite powder were dissolved in 350 mL of concentrated sulfuric acid (H₂SO₄) in a round bottom flask. The mixture was stirred well for 15 min to ensure uniform dispersion of graphite. The flask was allowed to rest in an ice bath to maintain a low temperature, while phosphoric acid (H₃PO₄) was dropped carefully. Afterwards, 18.0 g of potassium permanganate (KMnO₄) was introduced to the solution under continuous stirring and controlled heating, to facilitate the oxidation process. The reaction temperature was carefully regulated,

initially held at 35°C for 60 min, increased to 60°C for 4 hrs, and then reduced to 45°C for an extended period of 48 hrs. Upon completion, the mixture was gradually diluted with 300 mL distilled water, followed by the addition of 20 mL hydrogen peroxide (H₂O₂). The solution color shifted from dark brown to greenish brown, indicating reduction of excess permanganate. The diluted solution was then transferred to a 2 L graduated cylinder and topped up with distilled water, left to settle for 24 hrs to allow for sedimentation. The washing process, involving decantation and refilling with fresh distilled water, was repeated over two successive days. Finally, the suspension was centrifuged and extensively washed with distilled water until the pH of the solution reached near neutral. The purified GO was then spread out on petri dishes and air-dried over several days, yielding GO in the form of thin sheets as presented in **Figure 7**.

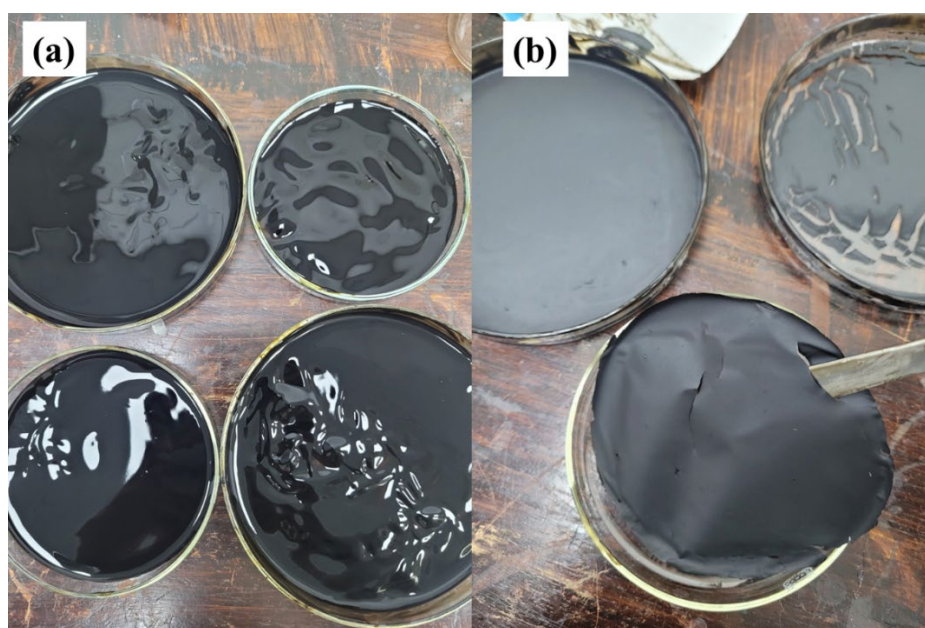


Figure 6. The GO nanosheets **(a)** before and **(b)** after drying.

3.3 Instrumentation

For hydrothermal synthesis, a stainless-steel hydrothermal autoclave reactor (50 mL) with a polytetrafluoroethylene (Teflon) lining tube was used. Characterization of the synthesized materials was performed using the following analytical instruments: The XRD patterns of the synthesized materials were analyzed by Phillips X'pert Pro powder X-ray diffractometer, with Cu K α radiation source ($\lambda = 0.15431$ nm). The Fourier-transform infrared (FTIR) spectroscopy, conducted with a Perkin Elmer spectrophotometer, was utilized to study the chemical bonding within the material. The samples were prepared with potassium bromide (KBr) pellets and a hydraulic press. Morphological and structural studies have been conducted using Thermo Scientific Quattro SEM, coupled Energy Dispersive X-ray (EDX) spectrometry for elemental analysis.

3.4 Application

The selected composites were evaluated for their efficacy in humidity sensing and as catalysts in the water-splitting reaction. The method, data collection, and setup of the experiments is discussed thoroughly below.

3.4.1 Assembly and measurement of humidity sensor

Humidity sensing measurements were accomplished by compressing the preferred nanomaterial into pellets by a hydraulic press and coating both sides of the pellets with a conductive silver paint as shown in **Figure 8**. The electrical measurements were executed by an LCR meter (Agilent E 4980A) at room temperature. The humidification process involved the

use of both saturated deionized water and NaOH solutions, with the measurements performed in a sealed environment.

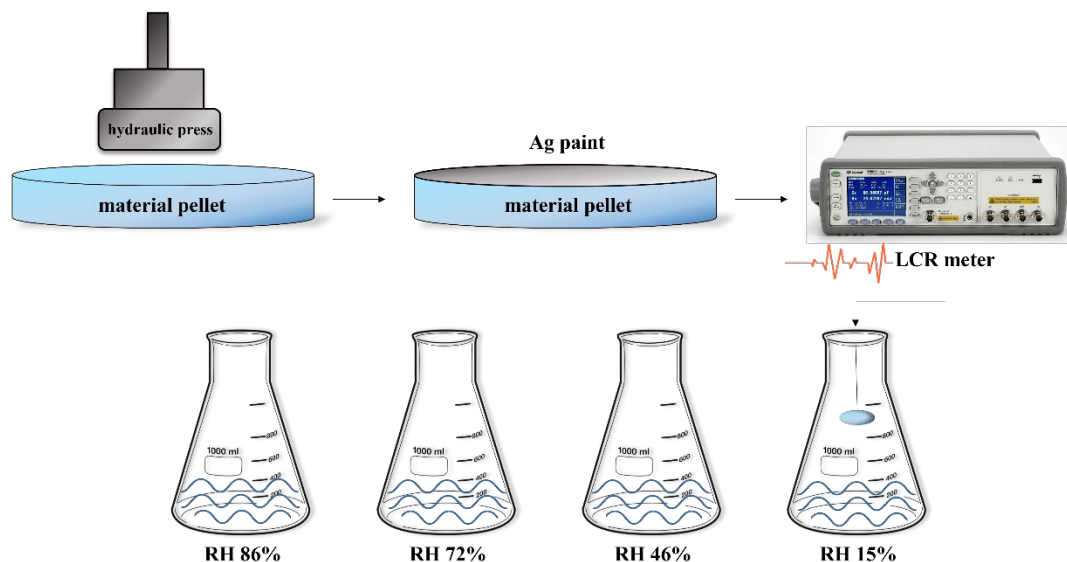


Figure 7. Schematic of the humidity sensing measurements.

3.4.2 Measurements for the HER/OER

A standard three-electrode setup was used to conduct electrochemical measurements using Zahner IM6EX electrochemical workstation (Germany). The saturated calomel electrode (SCE) reference electrode was utilized as the reference electrode, while platinum (Pt) foil served as a counter electrode. For typical measurements, 1 mg of the catalyst was dispersed in a mixture of 1 ml and 50 μ l of Nafion solution and ultrasonicate for 25 min, small aliquots (10-20 μ l) were dropped onto the glassy-carbon disk with the help of a syringe and allowed to dry at room temperature. Linear sweep voltammetry (LSV) was performed to investigate the

electrocatalytic activity of samples, proceeding at a scan rate of 1 mVs⁻¹ in an acidic medium of 0.5 M H₂SO₄. To ensure the absence of oxygen residues, nitrogen gas was purged through the solution one hour prior to the measurements. All electrochemical potentials reported in this work are referenced to the saturated calomel electrode (SCE) reference electrode. The zero-volt potential vs SCE was calibrated in the same electrolyte by measuring HER currents on a Pt foil electrode, whereby the potential at zero current corresponds to 0 V vs SCE.

3.5 Statistical Methods

To ensure clarity in the presentation of experimental results, data collected from various instruments were recorded as an xy data format. Subsequently, the dataset was analyzed and graphically represented using OriginPro 9, a widely recognized graphing and data analysis software extensively utilized in scientific research for its high analytical capabilities. Additionally, to enhance understanding of some synthesis processes employed in this study, schematic diagrams were designed using Microsoft Office PowerPoint. These diagrams serve to visually express synthesis methods, thereby complementing the quantitative data analysis.

Chapter Four:

Findings and Discussion

4. Chapter Four: Findings and Discussion

This chapter presents and critically evaluates the results obtained in the duration of the study. The chapter describes the characterization methods employed to assess the properties of the synthesized materials. The characterization of the prepared materials served as a benchmark to validate or reject the method of synthesis. Subsequently, materials demonstrating decent characterization results will be utilized in the application of humidity sensing and water splitting reaction, where a comprehensive discussion on the performance of selected materials in these applications is provided.

4.1 Material Characterization

The synthesized nanomaterials underwent a thorough characterization process employing XRD, FTIR, SEM, and EDX. Each technique had been selected for its capability to reveal critical aspects of the materials. The XRD was utilized to study the crystalline structure and phase composition, while FTIR provided a spectral fingerprint for identifying the functional groups and understanding the chemical bonding in the material. The SEM was used to visualize the nanomaterials' morphology, revealing surface features and topography at a micro to nanoscale resolution. Lastly, EDX was utilized to study the elemental composition and confirm the presence of intended dopants or impurities (Mourdikoudis et al., 2018). The selection of these analytical techniques aligns with previously established protocols for the characterization of nanomaterials (H. Bai, Maxwell, Kordes, & Balk, 2023; Ghazal, Mirzaee, Darroudi, Sabouri, &

Khadempir, 2024; Phua et al., 2023; Pimalai, Putnin, & Bamrungsap, 2025).

4.1.1 Characterization of g-C₃N₄

The crystal structure and the chemical bonding of as prepared g-C₃N₄ were studied by XRD and FTIR. The main diffracted peak corresponds to (002) plane found at 27.6° for g-C₃N₄ is indicative of the interlayer stacking of conjugated aromatic rings as shown in **Figure 9a**. The calcination process at high temperatures is known to promote the formation of these graphitic layers (Tarighati Sareshkeh et al., 2023). The FTIR spectroscopy was used to investigate the functional group analysis of synthesized g-C₃N₄ in **Figure 9b**. The g-C₃N₄ showed a strong broad band from 3100-3600 cm⁻¹. This can be attributed to N-H stretching originated from NH₂ terminals in the g-C₃N₄, and the O-H stretching that results from the introduction of hydroxyl groups (OH and COOH) due to the exfoliation of g-C₃N₄ with nitric acid (X. Zhu et al., 2022). The peaks at 1411.9 cm⁻¹ and 1728.2 cm⁻¹ can be ascribed to C=N stretching mode. The triazine ring in g-C₃N₄ induced a typical vibrational mode at 895 cm⁻¹ and 810.1 cm⁻¹, while the peaks at 1249.9 cm⁻¹ and 1319.3 cm⁻¹ are for the aromatic C-N stretching mode, were the peaks overlapped with the C=N stretching mode (Adhikari et al., 2015).

The morphology and elemental composition of g-C₃N₄ were studied using SEM and EDX. In **Figure 9c**, the nanosheets of g-C₃N₄ appears to have a flaky, layered texture. The graphs in **Figure 9d** display the EDX spectra of g-C₃N₄. **Figure 9d** exhibits prominent peaks for carbon (C) and

nitrogen (N), which are the primary constituents of $\text{g-C}_3\text{N}_4$. The absence of other peaks suggests the high purity of the material.

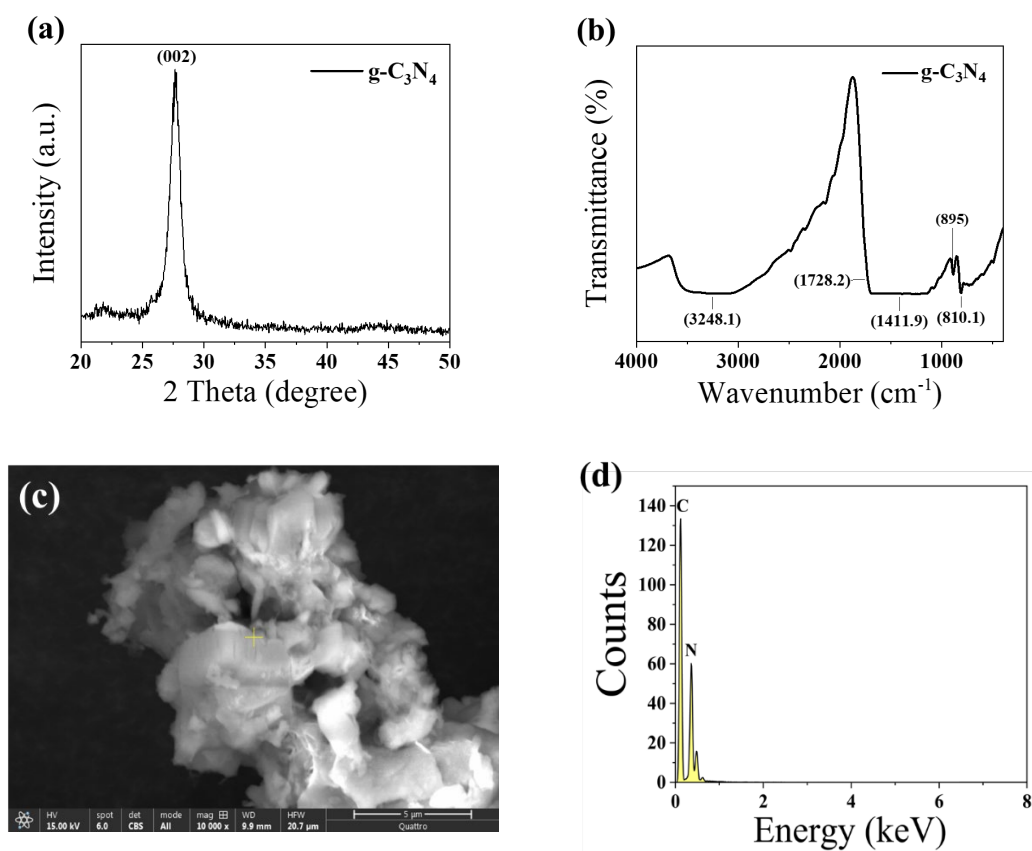


Figure 8. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for pristine $\text{g-C}_3\text{N}_4$.

4.1.2 Characterization of WO₃

The XRD analysis demonstrated the formation of monoclinic WO₃ nanoparticles. The XRD spectrum, displayed in **Figure 10**, exhibits distinct peaks at 2θ values of 23.1° , 23.7° , and 24.3° , corresponding to the (002), (020), and (200) crystallographic planes, respectively. These peaks are characteristic indicators of the monoclinic phase of WO₃, confirming the material's crystal structure as previously reported (Matalkeh et al., 2022).

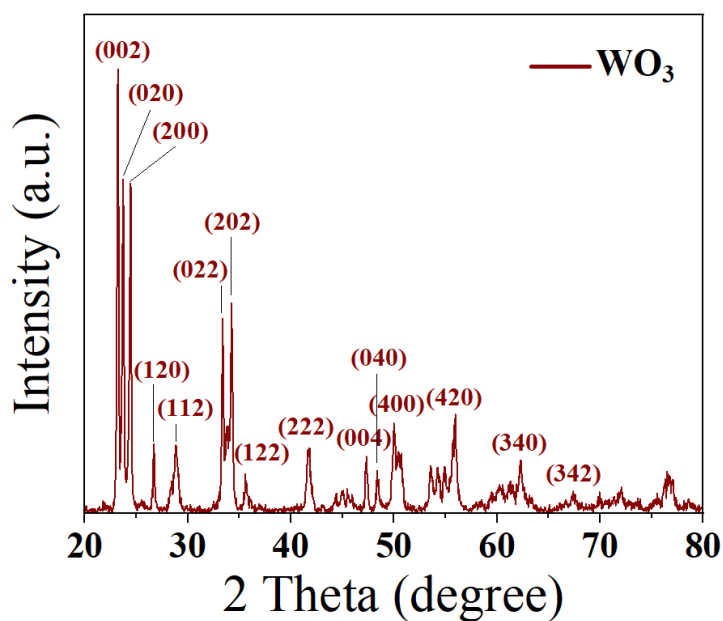


Figure 9. The XRD pattern for bare WO₃ nanoparticles.

4.1.3 Characterization of WO₃@g-C₃N₄

The crystal structure and the chemical bonding of WO₃@g-C₃N₄ were studied by XRD and FTIR as shown in **Figure 11a & b**. The XRD pattern for WO₃@g-C₃N₄ in **Figure 11a** displays all the main diffraction planes of WO₃ as shown in **Figure 10**. Moreover, an increase in the intensity of the (112) peak of plane for WO₃ at 27.6° is observed, as it overlapped with the main characteristic (002) peak of plane for g-C₃N₄.

Moreover, the W-O-W and the W-O-H stretching modes are observed at 1651.1 cm⁻¹ and 779.2 cm⁻¹ in **Figure 11b**, respectively, consistent with WO₃ known characteristic peaks. The characteristics peaks of g-C₃N₄ in the WO₃@g-C₃N₄ align with the original peaks in the pristine g-C₃N₄ spectrum. Notably, the spectral broadening observed may be indicative of surface adsorbed water molecules, as proposed in literature (Yesmin, Hussain, Dasgupta, & Dhar, 2023).

The SEM analysis presented in **Figure 11c**, distinctly reveals the spherical structure of the WO₃@g-C₃N₄ nanocomposite. A granular and porous morphology was also observed, which is typical for composite materials that combine nanoparticles with a layered substrate (J. Bai & Zhou, 2014). The EDX spectra, as shown in **Figure 11d**, confirms the elemental composition, displaying peaks representative of tungsten (W) and oxygen (O), and identifying the presence of C. The EDX data suggested a relatively low concentration of C and N in the analyzed region, which accounts for the absence of N signal in the spectrum (Goldstein et al., 2018). The characterization techniques collectively affirm the successful synthesis of the WO₃@g-C₃N₄ nanocomposite.

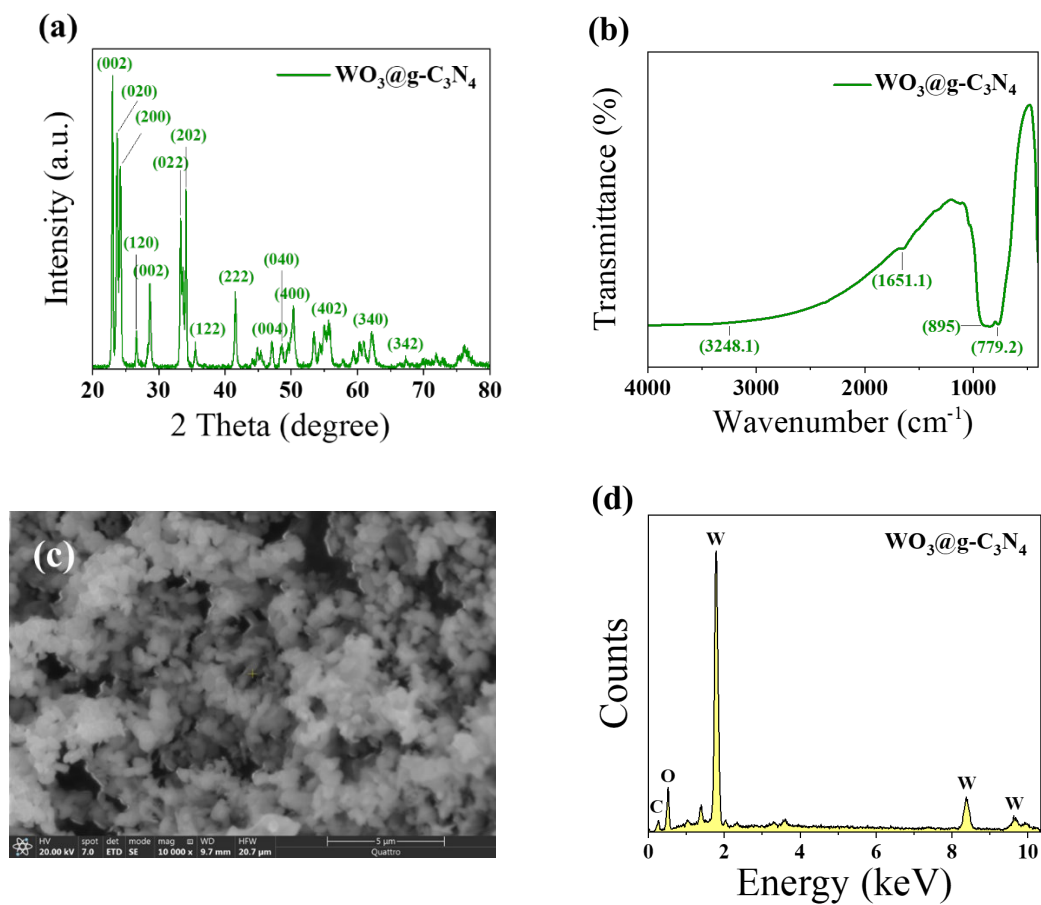


Figure 10. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for $\text{WO}_3@\text{g-C}_3\text{N}_4$.

4.1.4 Characterization of Ag/WO₃@g-C₃N₄

The crystal structure and the chemical bonding of Ag/WO₃@g-C₃N₄ were studied by XRD and FTIR as shown in **Figure 12a & b**. The XRD pattern for Ag/WO₃@g-C₃N₄ in **Figure 12a**, exhibited prominent diffraction peaks consistent with both WO₃ and g-C₃N₄. The absence of distinct Ag peaks is likely due to the minimal concentration of Ag within the composite (Moradi, Yousefi, Adelpour, & Sadeghi, 2023).

The FTIR of the Ag/WO₃@g-C₃N₄ nanocomposite, presented in **Figure 12b**, showed well-defined peaks for WO₃ and g-C₃N₄. The peaks remain unchanged upon Ag doping. The absence of characteristic Ag peaks is due to the low content of Ag, and is in agreement with findings in literature Ag (Amarnath et al., 2024; Qi et al., 2019).

The presence of Ag was confirmed by morphological and elemental composition analysis accomplished by SEM and EDX in **Figure 12c & d**. Compared to the WO₃@g-C₃N₄ in **Figure 11b**, the presence of silver appears to contribute to a more pronounced particulate definition, where the Ag nanoparticles can potentially enhance the porosity and create additional active sites (Oh, Lok, Veksha, Giannis, & Lim, 2018). Additionally, in **Figure 12d**, the main peaks observed in the EDX analysis are C, N, W, O, and Ag, confirming the synthesis of the Ag/WO₃@g-C₃N₄.

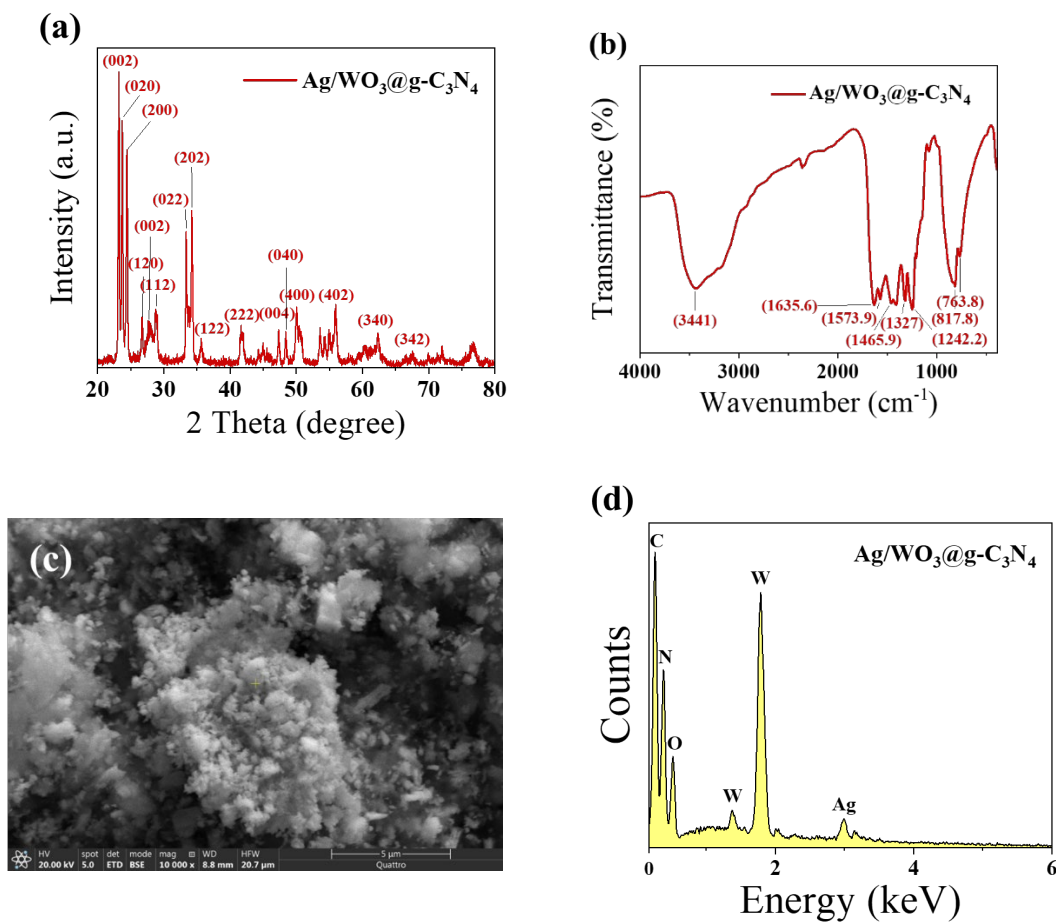


Figure 11. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for Ag/WO₃@g-C₃N₄.

4.1.5 Characterization of Co@g-C₃N₄

The crystal structure and the chemical bonding of Co@g-C₃N₄ were studied by XRD and FTIR as shown in **Figure 13a & b**. The XRD pattern in **Figure 13a** demonstrates a reduced intensity of the main characteristic (002) peak of plane for g-C₃N₄ may indicate a decrease in crystallinity or an increase in lattice defects which can be ascribed to the distortion of g-C₃N₄ crystal cell after Co doping (Chu, Younis, & Li, 2012; Pan et al., 2022). Moreover, no detectable peaks were observed corresponding to

metallic cobalt or cobalt oxide (Sanad, Taha, Helal, & Mahmoud, 2023), which could be due to low concentration of cobalt doping and the homogenous distribution across the g-C₃N₄ sample (C. Han et al., 2014; Wu et al., 2018). The low intensity peaks appearing in the Co@g-C₃N₄ pattern corresponds to Co peaks (Rahimi-Nasrabadi, Naderi, Karimi, Ahmadi, & Pourmortazavi, 2017).

For the FTIR of Co@g-C₃N₄ sample in **Figure 13b**, some key differences from pristine g-C₃N₄ are observed which are attributable to cobalt doping. Notably, additional peaks emerged at 547.8 cm⁻¹ and 632.7 cm⁻¹, which corresponds to Co-O vibrational mode, suggesting the successful introduction of cobalt into the lattice (Sanad et al., 2023). The characteristic peaks at 3441 cm⁻¹ and 802.4 cm⁻¹ assigned to N-H stretching and the triazine ring of g-C₃N₄, respectively, appear to be shifted than the original undoped material. The shifts may be due to changes in the electronic environment around the nitrogen atoms caused by cobalt doping. The peaks at 1728.2 cm⁻¹ and 1589.3 cm⁻¹ corresponds to C=O bending and carboxylate absorption band, respectively (X. Zhu et al., 2022). The 1249.9 cm⁻¹ and 1319.3 cm⁻¹ peaks for the aromatic C-N stretching mode are clearer and more visible in the FTIR spectrum for Co@g-C₃N₄ than pristine g-C₃N₄. The vibrational modes arising from the g-C₃N₄ are relatively strong, therefore the peaks appear to be overlapping in some regions.

On the other hand, **Figure 13c** clearly displays a nanorod-like structure of the Co@g-C₃N₄ in the SEM analysis. The nanorods appear to have a uniform diameter. The even distribution of the nanorods suggests a controlled synthesis process, where the particles were well dispersed by

sonication during synthesis (Glaubitz, Rothen-Rutishauser, Lattuada, Balog, & Petri-Fink, 2022).

The EDX spectra of Co@g-C₃N₄ in **Figure 13d** shows peaks for C and N as well as a peak for Co which is not present in **Figure 9d**. This confirms the successful modification of g-C₃N₄ with cobalt in the Co@g-C₃N₄ composite. In addition, the presence of a small O peak in **Figure 13d** may be attributed to some surface oxidation of Co.

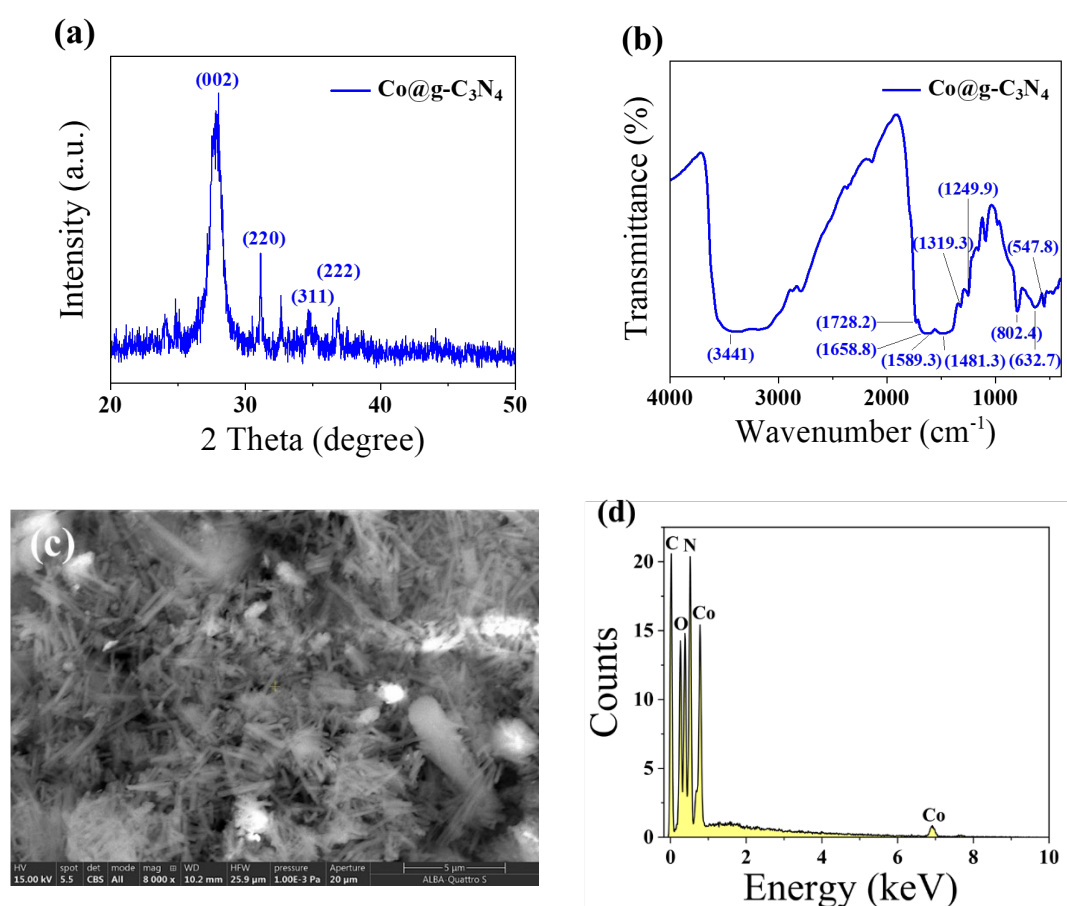


Figure 12. Characterization results of the (a) FTIR, (b) XRD, (c) SEM and (d) EDX for Co@g-C₃N₄.

4.2 Selection of the Material

The selection of suitable materials for applications in humidity sensing and water splitting reaction prioritizes facile synthesis, good electrical conductivity, efficiency, water sensitivity, and chemical stability (Rittersma, 2002). The g-C₃N₄ in particular, exhibits high water sensitivity, and so was considered as the control material (Gao et al., 2022). Therefore, composites with g-C₃N₄ were pursued. However, the g-C₃N₄ lacked good conductivity and chemical inertness, requiring metallic doping to enhance performance (Luo, Yan, Zheng, Xue, & Pang, 2019).

The successful synthesis and doping of Co in g-C₃N₄, as well as WO₃ in g-C₃N₄, and Ag doped WO₃@g-C₃N₄, have been verified through comprehensive characterization (**section 4.1.1 - 4.1.5**). These composites are particularly interesting since the literature on their use in humidity sensing and water splitting reaction remains unexplored –to the best of our knowledge– , providing a novel research route. For comparative purposes, the performance of WO₃@g-C₃N₄ was also examined.

The material selection process highlights a strategic approach, where a balance between previously established knowledge and the potential for innovation is taken into consideration, while aligning material properties with specific application requirements.

4.3 Application

In accordance with the findings presented in **section 4.2** and **section 4.3**, $\text{g-C}_3\text{N}_4$, $\text{Co@g-C}_3\text{N}_4$, $\text{WO}_3\text{@g-C}_3\text{N}_4$, and $\text{Ag/WO}_3\text{@g-C}_3\text{N}_4$ were utilized for humidity sensing applications. Additionally, both the $\text{g-C}_3\text{N}_4$ and $\text{Co@g-C}_3\text{N}_4$ were applied for the water splitting reaction.

4.3.1 Humidity Sensing Performance of Selected Materials

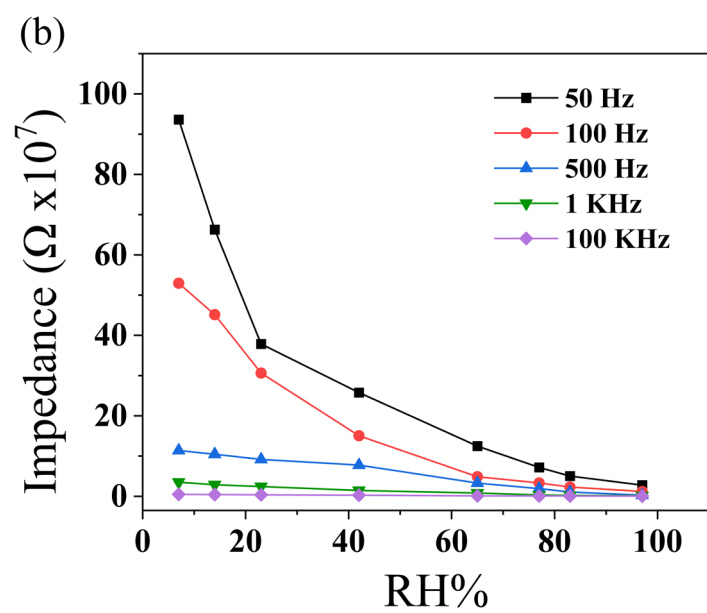
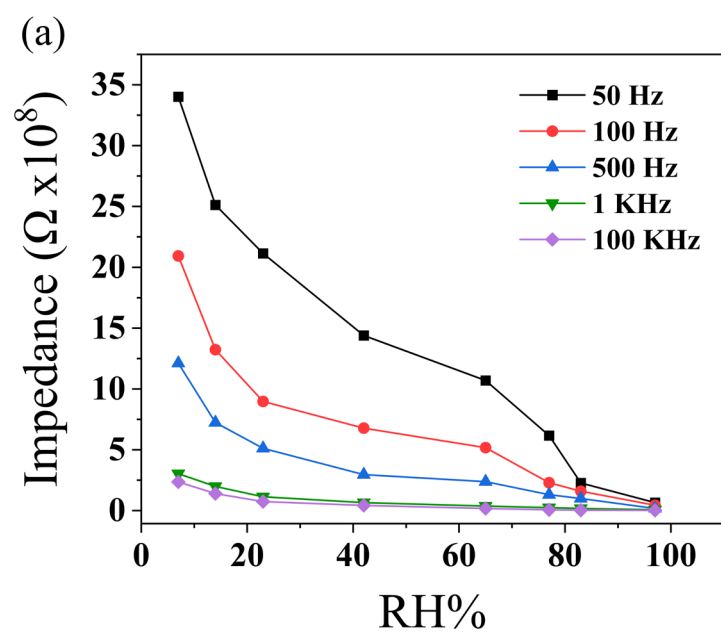
The as-prepared materials were tested for their humidity sensing capabilities at various humidity levels. The measured impedance of the four sensors, $\text{g-C}_3\text{N}_4$, $\text{Co@g-C}_3\text{N}_4$, $\text{WO}_3\text{@g-C}_3\text{N}_4$, and $\text{Ag/WO}_3\text{@g-C}_3\text{N}_4$, upon exposure to various RH levels was shown in **Figure 18**. The applied operational frequencies from 50 Hz to 100 KHz were chosen. The impedance of the four devices decreases with the increasing RH at fixed operation frequency. The adsorption of water molecules could promote ionic conductivity and enhance the dielectric constant of the sensing film. It is noteworthy to mention here that the sensing response of all the devices were more efficient for lower frequencies than at higher frequencies and the largest impedance variation upon exposure to 7%–94% RH was recorded at 50 Hz for $\text{Co@g-C}_3\text{N}_4$, $\text{WO}_3\text{@g-C}_3\text{N}_4$, and $\text{Ag/WO}_3\text{@g-C}_3\text{N}_4$. The sensitivity of $\text{Co@g-C}_3\text{N}_4$ and $\text{WO}_3\text{@g-C}_3\text{N}_4$ was far better than $\text{g-C}_3\text{N}_4$ at all frequencies as shown in **Figure 18 a&b**. The lowest impedance of all frequencies was recorded for $\text{Ag/WO}_3\text{@g-C}_3\text{N}_4$ in **Figure 18d**. Overall, at higher frequencies, the response to impedance change becomes less for all sensors. At higher frequencies, the water molecules are hardly dissociated thus affect the conductivity of material, and therefore, its impedance. The optimized frequency based on the former tests was chosen

to be 100 Hz, as the data is more linear and has decent sensitivity towards the change in impedance response for the sensors. Therefore, all subsequent measurements were carried out at 100 Hz for all the sensing devices.

The hysteresis is an important characteristic to express the adsorption and desorption process of the sensors. The switching RH test was performed by exposing the sensors to different humidity levels from low to high and then conversely from high RH to low RH. The hysteresis of the four sensors is shown in **Figure 19a**. An enlarged scale of the $\text{WO}_3@\text{g-C}_3\text{N}_4$ and $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$ hysteresis is shown in **Figure 19b**. The upper curves (black, blue, violet, and cyan) represent the increasing RH (adsorption) process, and the lower curves (red, green, purple, and turquoise) represent the decreasing RH (desorption) process. The variation in adsorption-desorption curves were found to be 23%, 15%, 21%, and 20% for $\text{g-C}_3\text{N}_4$, $\text{Co}@\text{g-C}_3\text{N}_4$, $\text{WO}_3@\text{g-C}_3\text{N}_4$, and $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$, respectively, as shown in **Figure 19a**. This indicates that the doped composites had highly reversible properties as compared to $\text{g-C}_3\text{N}_4$ and hence has greater implications for practical applications. The $\text{Co}@\text{g-C}_3\text{N}_4$ showed the highest reliability.

The $\text{g-C}_3\text{N}_4$ nanosheets have a strong affinity to water molecules. However, due to the poor conductivity of $\text{g-C}_3\text{N}_4$, the dissociation of water molecules to charged protons is relatively weak. This results in poor conductivity and sensitivity. Metal doping with Co, WO_3 , and Ag may facilitate the dissociation of water molecules adsorbed on the surface and enhance the material's conductivity. Overall, the $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$ exhibited the best humidity sensing performance, and good reversible

properties. The presence of Ag in the composite potentially creates localized regions of high conductivity, thus lowering impedance. The presence of silver may also aid in the adsorption and desorption processes of water molecules on the sensor surface.



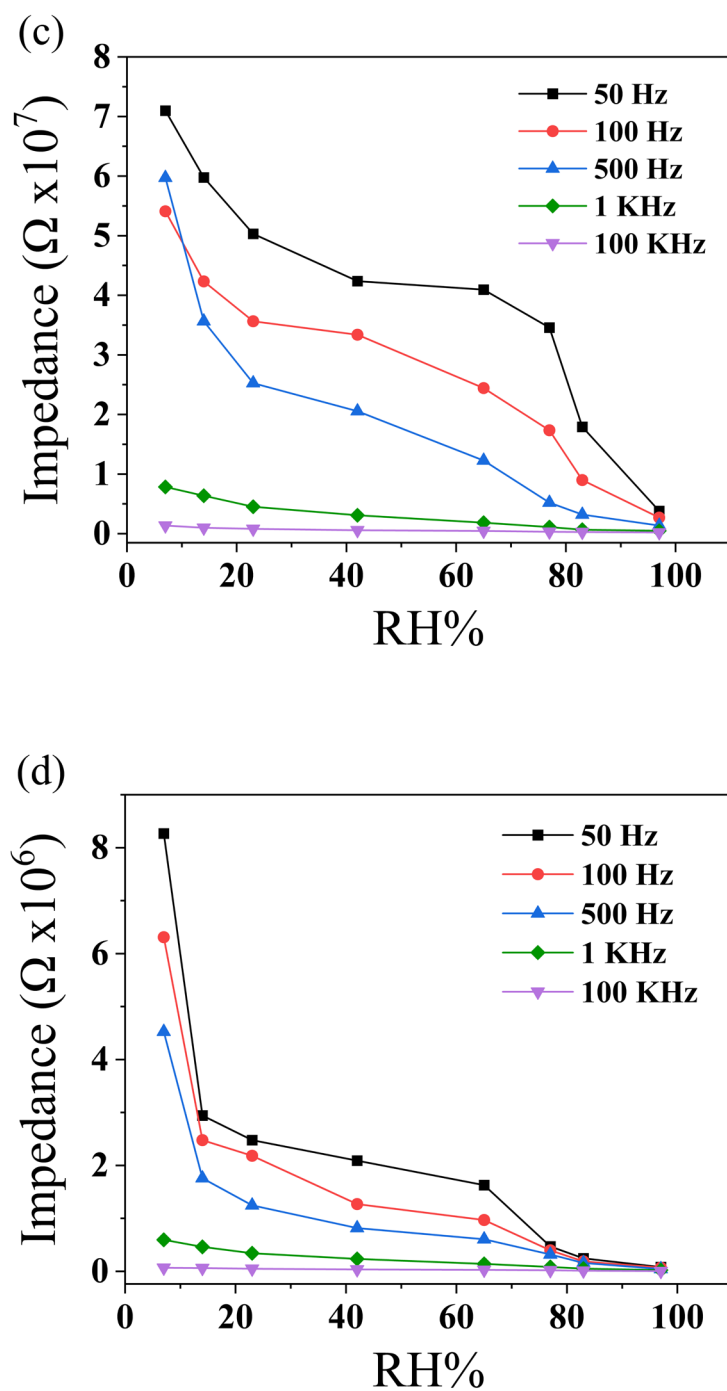


Figure 13. The measured impedance of the four sensors (a) $\text{g-C}_3\text{N}_4$, (b) $\text{Co}@\text{g-C}_3\text{N}_4$, (c) $\text{WO}_3@\text{g-C}_3\text{N}_4$, and (d) $\text{Ag/WO}_3@\text{g-C}_3\text{N}_4$, upon exposure to various RH levels.

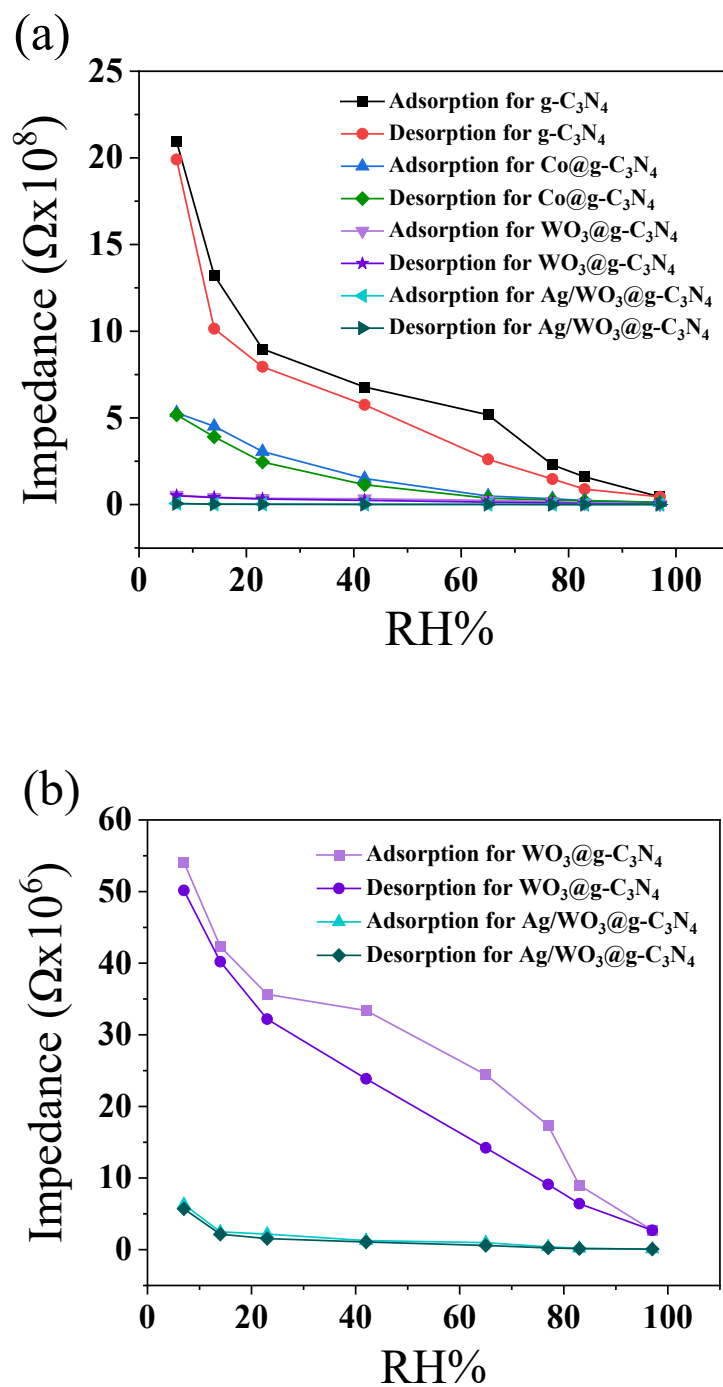


Figure 14. The hysteresis of (a) $\text{g-C}_3\text{N}_4$, $\text{Co@g-C}_3\text{N}_4$, $\text{WO}_3\text{@g-C}_3\text{N}_4$, and $\text{Ag/WO}_3\text{@g-C}_3\text{N}_4$ and (b) enlarged scale for $\text{WO}_3\text{@g-C}_3\text{N}_4$, and $\text{Ag/WO}_3\text{@g-C}_3\text{N}_4$.

4.3.2 Water Splitting Reaction

4.3.2.1 HER Performance of Selected Materials

Linear sweep voltammetry was conducted to investigate the electrocatalytic HER performance for g-C₃N₄, Co@g-C₃N₄, and Pt at a scan rate of 1 mV s⁻¹. The working electrodes were pre-stabilized for at least 30 CV cycles with the scan rate of 1 mV/s before measurement of polarization curves. The resulting polarization curve is shown in **Figure 20a** where the current density (J) in mA cm⁻² was plotted against applied sweeping potential. Note that the onset of HER is analyzed as the potential at which the observed current initially begins to deviate from background current. The polarization curve for g-C₃N₄ (black curve) has an onset potential (potential with current density > 1 mA/cm²) of ~ 0.48 V and shows a gradual increase in the current density with an increase in the overpotential. The values of overpotential (η_{10}), potential at which current density increases over 10 mA/cm², for g-C₃N₄ sample is ~0.49 V. The Co@g-C₃N₄ sample decreases onset potential to 0.42 V as well as the value of η_{10} to ~0.41 V (blue curve), respectively. The superior performance of Pt electrode is expected given it is a pure metal which has a very small binding energy for H⁺.

To obtain a better understanding of the kinetics and the mechanism of the reaction, the Tafel plots were calculated, where the applied potential (V) was plotted against the log of current density (J) as shown in **Figure 20b**. Smaller Tafel slopes are more convenient, as they represent faster reaction kinetics and favorable HER process. The Tafel slope of pristine g-C₃N₄ was recorded as 91 mV dec⁻¹, representing relatively slow kinetics for the HER process. The superior kinetics of the Co@g-C₃N₄ was evident

as the Tafel slope decreased to 64 mV dec^{-1} , and surpassed Pt at 131 mV dec^{-1} . Interpretation of the Tafel slope determines the mechanistic pathways during the HER process. Under acidic conditions, the Tafel slope of Co@g-C₃N₄ predicts the Volmer-Heyrovsky mechanism. The reaction of adsorbed hydrogen with an electron and a proton to generate hydrogen gas is the rate-determining step in Co@g-C₃N₄ as shown in **Figure 21**. The addition of metallic Co in g-C₃N₄ has been proven to enhance charge transfer, and increase the efficiency of the HER catalysis. Overall, the activity of the Co doped g-C₃N₄ toward the HER is inferior to the state-of-the-art catalysts. However, it is quite advantageous in that it is precious-metal free and is based on a low-cost synthetic route with water as the solvent.

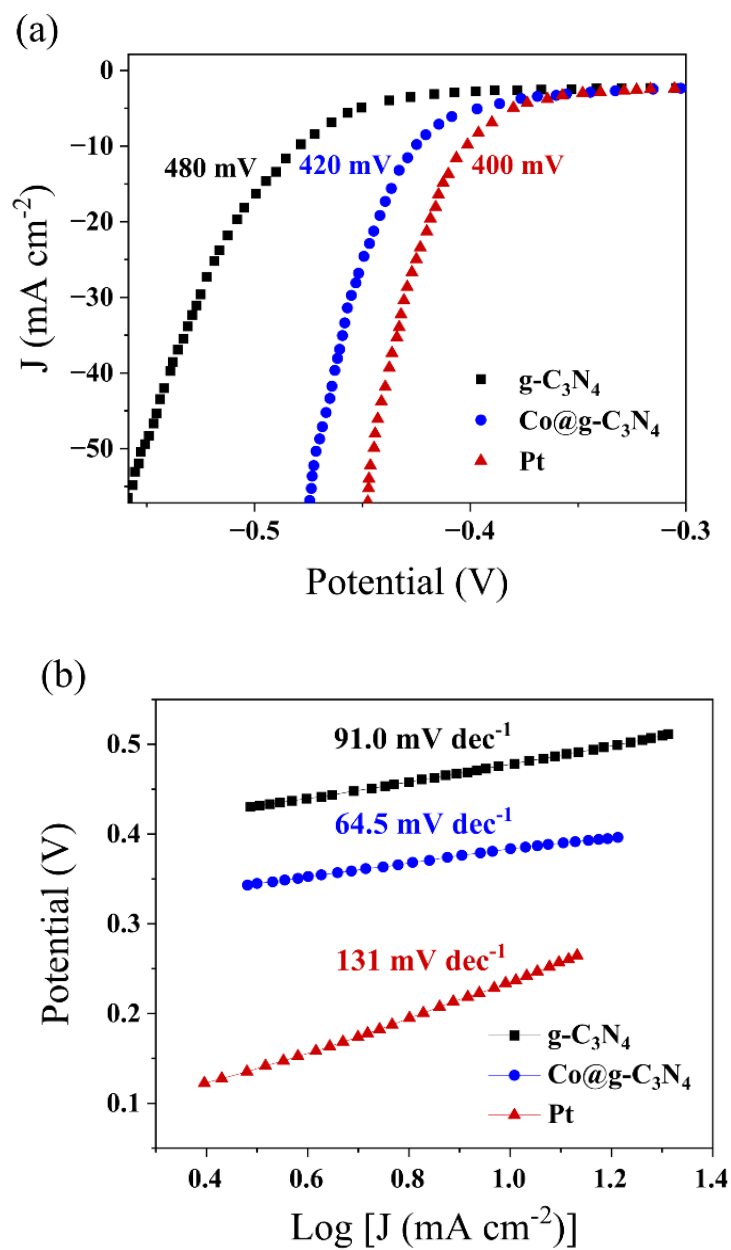


Figure 15. (a) HER polarization curve and (b) Tafel slope for the three electrocatalysts g-C₃N₄, Co@g-C₃N₄, and Pt.

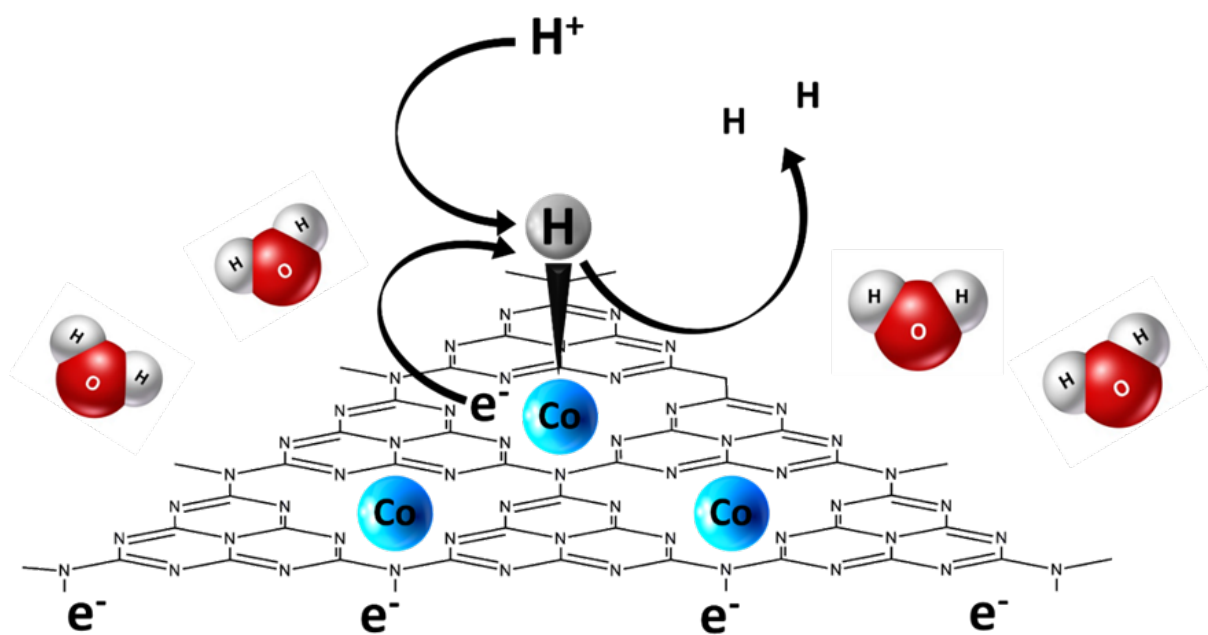


Figure 16. The Volmer-Heyrovsky mechanism followed by Co@g-C₃N₄ for the HER.

4.3.2.2 OER performance of selected materials

The performance of pristine g-C₃N₄ and Co@g-C₃N₄ towards the OER was accomplished by LSV at a scan rate of 1 mV s⁻¹ in acidic condition. The resulting anodic polarization curve is shown in **Figure 22**, where the current density (J) in mA cm⁻² was plotted against applied sweeping potential. The working electrodes were pre-stabilized for at least 30 CV cycles with the scan rate of 1 mV/s before measurement of polarization curves. Similar to the HER, the onset of the OER was analyzed as the potential the observed current initially begins to deviate from background current. The polarization curve of g-C₃N₄ (black curve) displayed an onset of 1.607 V, while the polarization curve of Co@g-C₃N₄ (blue curve) showed a decreased onset to 1.505 V. Although both polarization curves increase in the current density as the potential increases, the Co@g-C₃N₄ is more energy efficient as it requires less onset to drive the reaction.

The η_{10} of the OER is calculated as the difference between the actual potential and the standard reduction potential of the OER (1.23V). Therefore, it is unrealistic for a catalyst to practically achieve the exact value of 1.23 V. The polarization curve of g-C₃N₄ displayed an η_{10} of 370 mV, which is an average value towards the catalysis of OER. Remarkably, the polarization curve for Co@g-C₃N₄ revealed a very low η_{10} of 270 mV, exceeding the catalytic performance of pristine g-C₃N₄.

The η_{10} of the Co@g-C₃N₄ was compared with various reported catalysts for the OER in **Table 1**. The Co@g-C₃N₄ demonstrated superior OER catalysis relative to g-C₃N₄ based material (Alduhaish et al., 2019;

Fan et al., 2023; Vignesh, Nam, & Kim, 2024; Wen et al., 2023; M. Zhu et al., 2019). Notably, the Co@g-C₃N₄ not only outperformed certain noble metal-based catalysts (Demir, Akbayrak, Önal, & Özkar, 2019; M. Fu et al., 2020; F. Wang et al., 2019), but also matched the performance of others, like cobalt layered double hydroxide with sub-nanometer platinum clusters (Pt/Co-LDH) (S. Zhang et al., 2020), and iridium-based catalysts (Kim et al., 2021; Lee et al., 2023). The Co@g-C₃N₄ remains with several practical advantages like its facile synthesis, low cost, and accessibility.

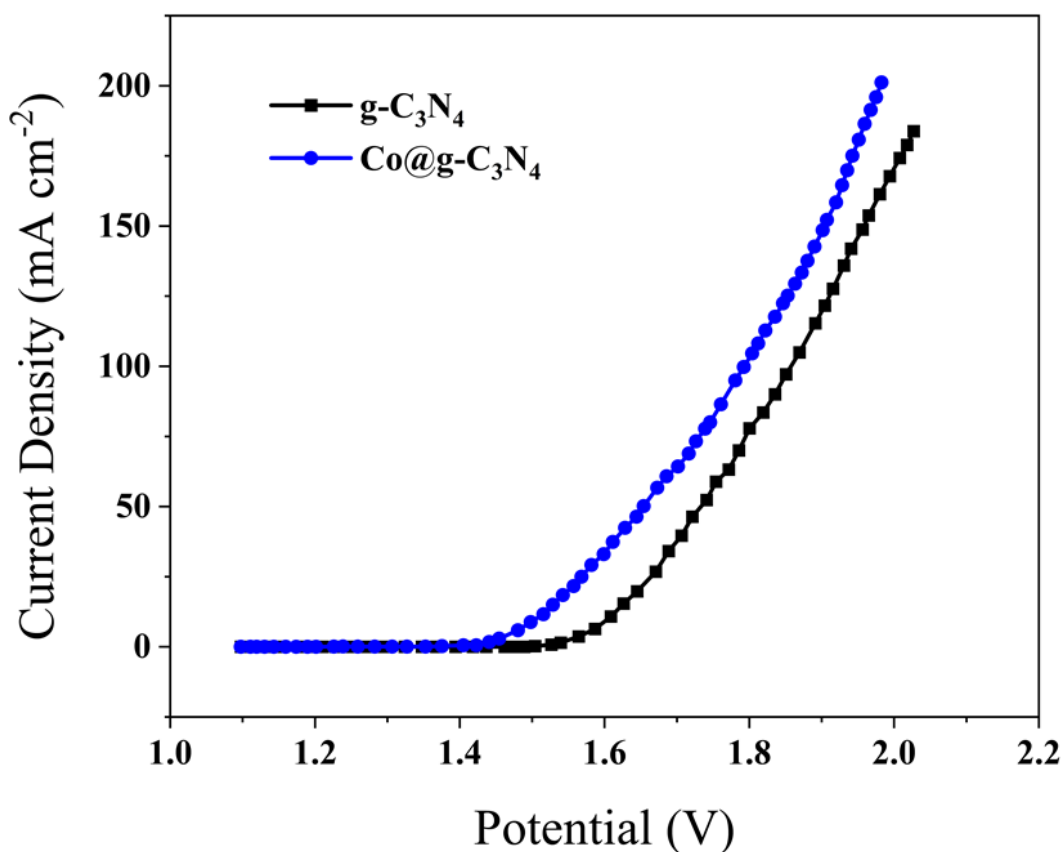


Figure 17. Anodic polarization curves for bare g-C₃N₄ and Co@g-C₃N₄.

Table 1. Comparison between the Co@g-C₃N₄ nanorods with g-C₃N₄ based materials, and precious-metals based materials reported in literature for the OER.

Catalyst	Overpotential (mV)	Reference
Co ₃ O ₄ /P-CN	320	(M. Zhu et al., 2019)
α -Fe ₂ O ₃ /g-C ₃ N ₄	425	(Alduhaish et al., 2019)
Co ₃ O ₄ / g-C ₃ N ₄	627	(Fan et al., 2023)
g-C ₃ N ₄ / Co ₃ O ₄ / α -Fe ₂ O ₃	359	(Vignesh et al., 2024)
MOF-derived Fe–NiS ₂ @ g-C ₃ N ₄	280	(Wen et al., 2023)
Ni-Fe embedded in Pt	333	(M. Fu et al., 2020)
Pt/Co-LDH	265	(S. Zhang et al., 2020)
RuO/CeO ₂	420	(Demir et al., 2019)
Ir/TiO ₂ –MoOx	290	(Kim et al., 2021)
IrOx/Zr ₂ ON ₂	255	(Lee et al., 2023)
Ni core and Au@Pd with rGO	520	(F. Wang et al., 2019)
Co@g-C ₃ N ₄	270	This work

4.4 Conclusion and Key Findings

To summarize, the research has successfully developed and characterized several low-cost g-C₃N₄ based nanocomposites, where the study focused on Co@g-C₃N₄, WO₃@g-C₃N₄, and Ag/WO₃@g-C₃N₄. The successful doping of Co, WO₃, and Ag in g-C₃N₄ was established by FTIR and XRD analysis. The SEM analysis confirmed the nanorods like morphology of Co@g-C₃N₄, and the spherical shape of WO₃@g-C₃N₄ and Ag/WO₃@g-C₃N₄. The research has demonstrated that doping g-C₃N₄ with metals such as Co, WO₃, and Ag enhances the electrical conductivity and catalytic efficiency of the g-C₃N₄ significantly. The four sensors were subjected to humidity sensing, where Ag/WO₃@g-C₃N₄ showed exceptional performance, and decent reversibility. Moreover, catalytic performance tests showed that Co doping succeeded in lowering the onset potential of g-C₃N₄ nanosheets towards the HER. The Co@g-C₃N₄ nanorods showed a mediocre activity, with a decent onset potential of 420 mV and low Tafel slope of 65.4 mV dec⁻¹. In addition, outstanding performance of the Co@g-C₃N₄ nanorods towards the OER, with a low overpotential of 270 mV and a good onset potential of 1.505 V at a current density of 10 mA/cm⁻² was recorded. As such, the reported performance of the Co@g-C₃N₄ nanorods is found to be superior to many other reported electrocatalysts, and is competitive with precious metal-based electrocatalysts. This work not only contributes to the advancement of material science in the field of catalysis and sensors but also proposes a pathway towards the development of environmentally friendly and cost-effective electrocatalysts.

4.5 Recommendations and Future Work

In the light of the findings presented in this thesis, several suggestions and future research is recommended as follows:

- Based on the results in section (humidity), the $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$ composite demonstrated promising potential for humidity sensing applications. Future investigations should extend to a comprehensive analysis of the $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$ long-term stability, selectivity, response, and recovery times.
- Given the enhanced conductivity attributed to the doping of Ag and WO_3 in the $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$, it would be of interest to investigate the utility of the $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$ for the HER and the OER.
- Exploring the real-time application of the $\text{Ag}/\text{WO}_3@\text{g-C}_3\text{N}_4$ composite in practical settings would be of interest, such as monitoring breath or soil humidity systems.
- It is advisable that any future studies involving the $\text{g-C}_3\text{N}_4$, to employ an exfoliation technique prior to the doping $\text{g-C}_3\text{N}_4$. Furthermore, the hydrothermal method, utilized for the synthesis of hybrid $\text{g-C}_3\text{N}_4$ composites, offers an environmentally friendly, rapid, and scalable approach, which is beneficial for large-scale applications.
- Advanced characterization techniques like the Transmission Electron microscopy (TEM) and the Brunauer-Emmett-Teller (BET) are essential for crucial information regarding the structural and surface properties of the materials. The TEM will determine the precise size of the particles, while the BET analysis will provide insights into surface area and porosity enhancement achieved through the several synthetic routes.

- Employ computational methods to model the electronic structure of the doped g-C₃N₄-based composites, and predict the properties of new composites prior to synthesis.
- The synthesized GO is of interest to be used in developing a series of composite containing it and investigating the various possible applications.

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الملخص

يبرز نيتريد الكربون الجرافيتي كمادة واعدة صديقة للبيئة لتطبيقات التحفيز والاستشعار بفضل خصائصها الفريدة. ومع ذلك، تعيق بعض العوائق مثل المساحة السطحية المحددة المنخفضة، وامتصاص الضوء غير الكافي، والتوصيل الكهربائي الضعيف، قابليتها للاستخدام على نطاق أوسع. تم تأسيس التشويب العنصري كنهج فعال لتعديل البنية الإلكترونية وفجوة النطاق لنيتريد الكربون الجرافيتي، مما يوسع بشكل كبير نطاق استجابتها للضوء. يتضمن هذا البحث تفاعلات صنع وتركيب العديد من المواد الكيميائية، واستخدام تقنيات وأجهزة حديثة لمعرفة خواص هذه المواد، والتأكيد على نجاح وفعالية طرق الصنع والتركيب. إن الأولوية في اختيار المواد التي ستستخدم في استشعار الرطوبة وتفاعل تفكيك المياه الكهربائي، يقع على المواد ذات الكفاءة العالية، مع توصيل كهربائي جيد، وحساسية عالية اتجاه مركب الماء، واستقرار كيميائي ممتاز، كما أنه من المهم أن تكون المواد ذات طرق تصنيع سهلة. يتضمن هذا البحث تصنيع نيتريد الكربون الجرافيتي النقي، ونيتريد الكربون الجرافيتي المشوب بالكوبالت، ونيتريد الكربون الجرافيتي المشوب بأكسيد التنجستن، ونيتريد الكربون الجرافيتي المشوب بأكسيد التنجستن والفضة، من خلال طريقتين بسيطتين في التصنيع: عملية التكليل المواد الصلبة، والتصنيع المائي الحراري. يتم تقييم أداء استشعار الرطوبة للمستشعرات الأربعة المذكورة سابقاً، عبر نطاق واسع من الرطوبة النسبية (٧٪ - ٩٤٪) بترددات مختلفة. تظهر مستشعرات نيتريد الكربون الجرافيتي المشوبة أداءً متفوقاً في استشعار الرطوبة ودقة أفضل مقارنةً بنيتريد الكربون الجرافيتي النقي، كما يتفوق نيتريد الكربون الجرافيتي المشوب بأكسيد التنجستن والفضة على المستشعرات الأخرى في أداء استشعار الرطوبة. علاوة على ذلك، يتم تقييم نيتريد الكربون الجرافيتي النقي و نيتريد الكربون الجرافيتي المشوب بالكوبالت لملائمتها كمحفزات لتفكيك الماء الكهربائي لإنتاج الهيدروجين (الشق الأول من تفاعل تفكيك الماء)، مما يمثل خطوة نحو خلايا الطاقة الفعالة. يعرض نيتريد الكربون الجرافيتي المشوب بالكوبالت أداءً محسناً مع جهد بدائي قليل (٤٢٠ ميلي فولت) ومنحدر تافل منخفض (٦٥.٤ ميلي فولت لكل عقدة) لتفاعل إنتاج الهيدروجين مقارنةً بنيتريد الكربون الجرافيتي النقي. بالإضافة إلى ذلك، يظهر نيتريد الكربون الجرافيتي المشوب بالكوبالت أداءً ممتازاً لتفاعل إنتاج الأكسجين (الشق الثاني من تفاعل تفكيك الماء)، حيث أظهرت النتائج جهد بداية قليل جداً (٢٧٠ ميلي فولت)، متفوقاً على العديد من المحفزات المذكورة في الأبحاث العلمية لتفاعل تفكيك الماء الكهربائي، كما تنافس هذه النتيجة المحفزات المصنوعة من المعادن النادرة والثرمينة.

جامعة البحرين

قسم الكيمياء



كلية العلوم

تصميم مركبات ثنائية وثلاثية نانوية فعالة من حيث التكلفة: تطبيقات كمواد محفزة وأجهزة استشعار للرطوبة

أطروحة مقدمة لاستيفاء جزء من درجة الماجستير في كيمياء البيئة

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