DEVELOPMENT OF MIXED CHALCOGENIDE HETEROSTRUCTURE NANOMATERIALS AS POTENTIAL PHOTOCATALYSTS FOR WASTE WATER TREATMENT

BY

CHIBUOKEM, MICHAEL ONYEDIKACHI
(14CC017713)
B.Sc. Industrial Chemistry Covenant University, Ota

A DISSERTATION SUBMITTED TO THE SCHOOL OF POSTGRADUATE STUDIES IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF MASTER OF SCIENCE (M.Sc.) DEGREE IN INDUSTRIAL CHEMISTRY IN THE DEPARTMENT OF CHEMISTRY, COLLEGE OF SCIENCE AND TECHNOLOGY, COVENANT UNIVERSITY.

JULY, 2022

ACCEPTANCE

This is to attest that this dissertation has been accepted in partial fulfilment of the requirements for the award of the degree of Master of Science in Industrial Chemistry in the Department of Chemistry, College of Science and Technology, Covenant University, Ota, Ogun State.

Mr. Taiwo B. Erewunmi
(Secretary, School of Postgraduate Studies) **Signature and Date**

Prof. Akan B. Williams
(Dean, School of Postgraduate Studies) **Signature and Date**

DECLARATION

I, **CHIBUOKEM, MICHAEL ONYEDIKACHI (14CC017713),** declare that this dissertation is a representation of my work, and is written and implemented by me under the supervision of Dr. Joseph A. Adekoya of the Department of Chemistry, College of Science and Technology, Covenant University. I attest that this dissertation has in no way been submitted either wholly or partially to any other university or institution of higher learning for the award of a masters' degree. All information cited from published and unpublished literature has been duly referenced.

CHIBUOKEM, MICHAEL ONYEDIKACHI

Signature and Date

CERTIFICATION

We certify that this dissertation titled "**DEVELOPMENT OF MIXED CHALCOGENIDE HETEROSTRUCTURE NANOMATERIALS AS POTENTIAL PHOTOCATALYSTS FOR WASTE WATER TREATMENT**" is an original research work carried out by **CHIBUOKEM, MICHAEL ONYEDIKACHI (14CC017713)** in the Department of Chemistry, College of Science and Technology, Covenant University, Ota, Ogun State, Nigeria under the supervision of Dr. Joseph A. Adekoya of the Department of Chemistry. We have examined and found the work acceptable as part of the requirements for the award of the degree of Master of Science (M.Sc.) in Industrial Chemistry.

Dr. Joseph A. Adekoya
(Supervisor) **Signature and Date**

Prof. Joseph. A. O. Olugbuyiro
(Head of Department) **Signature and Date**

Prof. Olayinka. T. Asekun
(External Examiner) **Signature and Date**

Prof. Akan B. Williams
(Dean, School of Postgraduate Studies) **Signature and Date**

DEDICATION

This dissertation is dedicated to God almighty.

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LIST OF ACRONYMS AND ABBREVIATIONS

C.A	Citric Acid
CTAB	Cetyl trimethyl ammonium bromide
GLY	Glycerol
LED	Light Emitting Diode
QD	Quantum Dots
SEM	Scanning Electron Microscopy
TAA	Thioacetamide
TEM	Tri-n-ortho phosphine oxide
TOP	Tri-north phosphine
TOPO	Transmission Electron Microscopy
XRD	X-ray Diffraction

ABSTRACT

Water pollution is one of the world's biggest issues today. Several methods of purification have been put in place to curb this issue and more are still being researched upon. Photo and electro catalysis are just two of the methods discovered that can be used in the purification of water. The synthesis of catalysts that could speed up the rate of these reactions are of utmost importance. Chalcogenide heterostructures have been researched upon over the last few years based on the interesting band gap that is within the semi-conductor band gap range (1-3 eV). In this research work, eight nanoparticles were synthesised by varying different reaction conditions. The band gap of the synthesized nanomaterials ranged from 1.93 to 2.86 eV. The chalcogenide heterostructures synthesised are Ag_8SnS_6/FeS, $AgSnS_2$/FeS and Cu_xSnSe_y/FeSe. This was confirmed by the XRD analysis that revealed the different phases; the orthorhombic phase and the cubic phase under different synthesis conditions. The TEM analysis also showed that the nanomaterials formed where mostly nanocubes and with a little bit of nanosheets ranging from 28.83 nm ± 1.67 nm to 134.03 nm ± 3.51 nm. The factors varied were the temperature, the mole ratio of the ternary to binary material and the caping agents. The two capping agents used were citric acid and glycerol. The co-precipitation method was used to synthesise the nanoparticles. FTIR and UV-Vis spectrophotometric analyses were carried out on the resulting nanoparticles. The FTIR showed that the nanoparticles had a good surface functionalization confirming the presence of functional groups like the hydroxyl (O-H) at 3396.16 cm^{-1} and carbonyl group (C=O) at 1712.31 cm^{-1}, while the UV-Vis which was measured over a range of 250 to 1000 nm showed broad absorbance over 420 to 800 nm confirming that the nanoparticles had some optical properties. This showed that the nanoparticles would have a good photocatalytic application.

Keywords: Chalcogenide, Nanoparticle, Photocatalyst, Heterostructure, Co-precipitation.