AKENTEN APPIAH-MENKA UNIVERSITY OF SKILLS TRAINING AND ENTERPREURAL DEVELOPMENT

SYNTHESIS, CHARACTERIZATION AND APPLICATION OF ACTIVATED CARBON NANOTUBES IN REMOVAL OF Pb, As, AND Cd IN GROUNDWATERS IN BEREKUM MUNICIPALITY OF GHANA

SAMUEL YEBOAH ANTEPIM

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BY

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A THESIS IN THE DEPARTMENT OF CHEMISTRY EDUCATION, FACULTY OF SCIENCE EDUCATION, SUBMITTED TO THE SCHOOL OF GRADUATE STUDIES, AKENTEN APPIAH-MENKA UNIVERSITY OF SKILLS TRAINING AND ENTREPRENEURIAL DEVELOPMENT, IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR AWARD OF THE MASTER OF PHILOSOPHY (CHEMISTRY EDUCATION)

SEPTEMBER 2025

DECLARATION

Student's Declaration

I hereby declare that this work is my own original research and any quoted or referenced material has been properly cited. This submission has not been presented in part or whole for any other degree at any university, and I adhere to zero tolerance for plagiarism

CANDIDATE'S NAME: SAMUEL YEBOAH ANTEPIM

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Supervisor's Declaration

I certify that this thesis was prepared and presented under my supervision in accordance with the established guidelines by Akenten Appiah-Menka University of Skills Training and Entrepreneurial Development University.

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DEDICATION

This work is dedicated to my family whose constant love, support, and encouragement have inspired me every step of the way

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ABBREVIATIONS

A/F	-	After
AAS	-	Atomic Absorption Spectroscopy
AC	-	Activated Carbon
ACNTs	-	Activated Carbon nanotubes
APHA	-	American Public Health Association
As	-	Arsenic
B/4	-	Before
Cd	-	Cadmium
CNTs	-	Carbon nanotubes
CSIR	-	Council for Scientific and Industrial Research
EU	-	European Union
FTIR	-	Fourier Transform Infrared Spectroscopy
FWCNT	-	Few-Walled Carbon Nanotubes
GS	-	Ghana Standard
MWCNTs	-	Multiwalled Carbon Nanotubes
Pb	-	Lead
RWESCK	-	Regional Water and Environmental Sanitation Center
SD	-	Standard Deviation
SEM	-	Scanning Electron Microscopy
SWCNTs	-	Single Walled Carbon Nanotubes
TREATM	-	Treatment
USEPA	-	United States Environmental Protection Agency
UV VIS	-	Ultraviolet-Visible Spectroscopy
WHO	-	World Health Organization
XRD	-	X-Ray Diffraction

ABSTRACT

Safe, convenient, and cost-effective method of treating water to meet the drinking water needs of human is the key responsibility of upcoming scientist. Heavy metal contamination in water poses significant global challenge affecting both developed and developing nations due to their detrimental effect and health injuries on human and other organisms in the ecosystem. Carbon nanotubes has received the attention of researchers in water treatment and has been detailed premeditated in salt removal, heavy metal removal, removal of cosmetics, removal of antibiotics, removal of potentially toxic elements, and microorganism removal in drinking water. This investigation synthesized carbon nanotubes from graphite powder to remove Pb, As and Cd from ground water. Characterization with SEM, FTIR, UV-VIS and XRD showed polydisperse distribution of carbon nanotubes with particle sizes between 0.0553 µm (55.3nm) to 4.480 µm (480nm). UV showed multiwalled carbon nanotubes and XRD suggested moderate crystallinity. Synthesized carbon nanotubes showed effective adsorbent for removal of selected metal with removal rates of 95.36%, 96.3% and 94.33% for Pb, As and Cd respectively. The adsorption of Pb was best described by the Lagmiur isotherm model whereas the Freundlich isotherm model provided a better fit for As and Cd adsorption, as evidenced by coefficient if determination and chi-square error analysis. The activated carbon nanotubes have exhibited effective adsorption capacity for heavy metal treatment in water with higher maximum adsorption capacity which shows effective treatment of large volumes of water with higher metal contamination. The study suggest that activated carbon nanotubes will be used for water treatment for public due to economic feasibility and environmental sustainability.

CHAPTER ONE

INTRODUCTION

This section provides relevant information on an investigation concentrates on the use of Activated Carbon Nanotubes in heavy metal removal in Berekun municipality, Bono region of Ghana. This part of the work covers background to the study, brief literature grounding, problem statement, research objectives, research questions, justification, significance of the study, scope as well as organization of the thesis.

1.1 Background of the Study

Water is vital for sustaining life, playing important role in nourishing ecosystems, supporting human health, and driving economic development. The importance of water extends beyond its use by living organisms to industrial, agricultural, and domestic purposes.

Clean and safe water is a basic human right and its shortage can have devasting effects on both human well-being and the environment. Effective management and sustainable use of water resources are crucial for building a healthy, prosperous and equitable society (Onuoha and Ogbo, 2022). Water purification efforts notwithstanding, developing countries face escalating portable water scarcity, resulting in the increased waterborne diseases and negative health impacts. Contamination of municipal water supplies due to human activities poses severe public health and environmental concerns globally (Onuoha and Ogbo, 2022). Industrialization and urban growth have escalated water contamination worldwide. Water bodies are degraded by human activities, with rivers being disproportionately affected by pollutants from domestic, industrial, agricultural and mining sources resulting in biological, physical and chemical changes to the water (Apau et al., 2022). Fecal contamination significantly impacts river water quality in many developing countries. Often, inadequate infrastructure and insufficient water treatment fail to keep pace with rapid population growth exacerbating water pollution (Apau et al., 2022). Massive amounts of freshwater are polluted annually by various contaminants, rendering it undrinkable (Amari et al., 2023). Water pollutants, including dyes, heavy metals, pesticides, microorganisms, and hydrocarbons, pose significant threats to aquatic life and human health (Chahar et al., 2023). Heavy metals like lead, cadmium, arsenic, mercury, and chromium are particularly hazardous due to their toxicity, persistence, and bioaccumulative properties. Exposure to these metals can damage soft tissues in organs such as the liver, kidneys, brain, and lungs, leading to health issues like anxiety, nerve damage, cancer, and behavioral disorders (Gyamfi et al., 2023). Even low concentrations of these metals can have devastating effects on ecosystems and human well-being.

Traditional water remediation methods, such as chemical precipitation, ion exchange, and membrane filtration, can be effective but are often limited by high costs, sludge production, and reduced efficiency at low contaminant levels. While various water treatment technologies have been developed, many require significant investment, making them inaccessible in developing countries. In contrast, adsorption has emerged as a simple, cost-effective, and efficient method for removing organic and inorganic pollutants from water (Tome et al., 2021; Shikuku and Mishra, 2021; Nyairo et al., 2022). Adsorption stands out as a viable solution due to its scalability, cost-effectiveness, and ability to perform well even at low pollutant levels.

Notably, carbon nanotubes have revolutionized various fields, including water remediation (Abed et al., 2023). Carbon-based nanomaterials, such as activated carbon, CNTs, fullerenes, graphene, and graphene oxide, have gained significant attention for removing heavy metals from water due to their high surface area, customizable functionality, and potential for recycling (Attri, 2020).

Activated carbon nanotubes (ACNTs) merge the benefits of activated carbon's porosity with the unique structure of CNTs. Activation enhances CNT surface area, introduces functional groups, and improves dispersibility in water, collectively elevating adsorption potential.

1.2 Problem Statement

The increasing industrialization has led to a scarcity of clean, potable water, disproportionately affecting developing countries. Water pollutants, including organic matter, bacteria, viruses, dyes, and heavy metals like lead, cadmium, zinc, nickel, arsenic, chromium, and mercury, pose significant health risks. Exposure to these heavy metals can cause severe health issues, such as cancer, kidney damage, hepatitis, miscarriages, anemia, and neurological disorders (Baby & Saifullah, 2019). Heavy metal contamination remains a dire challenge affecting both developed and developing countries. Removing heavy metal ions from water is crucial to protecting human health and preventing associated health issues. Regions with limited resources where conventional treatment is beyond budget due to high cost of treatment and ineffective at low concentrations. Various methods can remove toxic metal ions from water, including ion exchange, reverse osmosis, precipitation, filtration, biosorption, coagulation, and extraction. However, adsorption stands out as a top choice due to its cost-effectiveness, high efficiency, and ease of operation, particularly for removing trace levels of heavy metals. There's a growing need for water treatment methods that are not only effective but also energy-efficient, affordable, and environmentally friendly, ensuring safe drinking water without compromising human health (Onuoha and Ogbo, 2022).

Carbon nanotubes (CNTs) are a promising new generation of carbon-based adsorbents with customizable surface chemistry. They have been effectively used to adsorb metal ions, anionic contaminants, organic compounds, and biological pollutants from water (Li et al., 2002; Peng et al., 2005; Cho et al., 2011). CNTs have also shown potential in water softening (Maryam and Toraj, 2011). Their versatility in removing a wide range of pollutants, including heavy metals and organic compounds, makes them an attractive option for water treatment applications. While activated carbon is prevalent, its performance for certain heavy metals remains suboptimal. Carbon nanotubes (CNTs) have gained significant attention in water treatment due to their potential in various applications, including desalination, heavy metal removal, and oil-water separation (Li et al., 2022). CNTs offer high adsorption potential but face limitations like high hydrophobicity, aggregation, clustering, cost, and a dearth of active surface sites.

There's a critical need for novel, cost-effective adsorbents that can surmount these issues. ACNTs, especially those derived from sustainable precursors like graphite promise enhanced heavy metal removal. Nevertheless, integrated investigations on their synthesis, physicochemical characterization, and environmental performance are spread out particularly with locally sourced affordable materials.

Water contamination is a demanding global issue, with heavy metals posing stern health damages to human and ecosystems. Metals like Pb, Hg, As and Cd in water can cause severe health complications including cancer, organ failure, and neurological damage. Effective and efficient removal of these pollutants have become a critical. ACNTs have arisen as capable material designed for eliminating heavy metals in water

1.3 Research Objectives

1.3.1 Main Objective

The core objective of the investigation is to synthesize, characterize, and evaluate activated carbon nanotubes (ACNTs) for heavy metal removal in ground water.

1.3.2 Specific Objectives

1. Synthesize ACNTs from graphite powder.
2. Characterize ACNTs with scanning electron microscopy (SEM), Fourier Transform Infrared spectroscopy (FTIR), Ultraviolent-visible spectrometer (UV-Vis) and X-RAY Diffractometer (XRD) to determine physicochemical properties.
3. Assess the adsorption capability of ACNTs for certain heavy metals (e.g., Pb^{2+}, Cd^{2+}, Cr^{6+}) under varied contact time and quantity of ACNTs.
4. Investigate adsorption mechanisms through Langmuir and Freundlich isotherm modeling.

1.4 Research Questions

1. What synthesis method yields ACNTs from less expensive and readily available precursors?
2. How do structural and surface properties of ACNTs facilitate heavy metal adsorption?
3. What are the adsorption efficiencies of ACNTs on the selected heavy metals?
4. Which adsorption mechanisms predominate in removals of heavy metals like Physisorption, chemisorption, pore diffusion, complexation?

1.5 Significance of the Study

Carbon Nanotubes has been a central technology for drinking water and wastewater treatment in the field of science and technology. The exceptional properties of Carbon Nanotubes have made it fundamental part of modern science in terms of removal of contaminants in water which is difficult to be removed by the old system of water purification being used in the country. This research advances water treatment innovation by bridging activated carbon and CNT technologies. It offers:

- Novel, easy, and low-cost synthesis method to make ACNTs accessible for large scale water treatment.
- A detailed understanding of how activation and functionalization of ACNTs affect metal adsorption from water.
- Empirical data on adsorption mechanisms of selected heavy metals on ACNTs for design optimization.
- Comparative insights for stakeholders to make valuable comparison about the most sustainable treatment options for heavy metals removal.
- Critical information on long term performance and sustainability of ACNT-based treatment systems.

1.6 Scope of the Study

This work intentions to investigate laboratory synthesis of ACNTs and evaluate their performance in heavy metal removal from ground water. This work will concentrate on:

- Laboratory synthesis of ACNTs from graphite powder
- Characterization of ACNTs including structural and surface analysis using the following techniques: SEM, FTIR, UV Vis and XRD.

- Adsorption studies to optimize ideal conditions such as contact time, and optimal dosage of ACNTs for maximum adsorption efficiency.

1.7 Organization of the Thesis

This work is prearranged into five chapters with each directed towards specific aspect of the research. The general layout is as follows:

Chapter One: Introduction: This episode covers related data on water as one of the most essential commodities for life, contaminants and their sources, the importance of treating heavy metals in water and the potential of ACNTs as treatment solution. It clearly presents aims of the search and research inquiries, Significance and the scope and limitation of the work including laboratory synthesis and specific heavy metal.

Chapter Two: Literature Review: Detailed analysis of linked literature on aquatic contamination and heavy metals comprising of various sources water pollutants, effects, and regulatory standards of various metals. Conventional methods of metal remediation and adsorption. Synthesis methods, characterization, and adsorption studies relevant to ACNTs and heavy metal removal.

Chapter Three: Materials and Methods: Synthesis from graphite powder, characterization techniques to determine physiochemical properties of ACNTs including SEM, FTIR, UV-Vis and XRD, and adsorption study design use to assess the performance of ACNTs on heavy metals.

Chapter Four: Results and Discussion: Presentation of results from characterization, and adsorption studies. Discussion of the performance of ACNTs on selected heavy metals using isotherm modelling.

Chapter Five: Key findings including size synthesized ACNTs, adsorption capacity for each heavy metal for development of workable and effective skill for heavy metal removal in water. Conclusion of the study including the maximum removal mnbbbmm, capability of activated carbon nanotubes explained by adsorption models, recommendations, and direction for future study.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

This section enumerates comprehensive analysis of current works done on heavy metal removal using ACNTs. Recent advancements in research on ACNTs synthesis, characterization, and application will be discussed, highlighting the key findings and research gaps. Knowledge on water pollution, heavy metals, adsorption technologies, and carbon-based nanomaterials will be synthesized. This literature review aims to summary of state of knowledge on synthesis and characterization, identifying research gaps in existing literature on ACNTs on heavy metal removal will inform the materials and methods used in this work.

This section will dig deep into heavy metal pollution in water, conventional treatment methods, adsorption theories, carbon-based adsorbents, and the current state of research on activated carbon nanotubes (ACNTs)

2.2 Heavy Metals in Water Pollution

Heavy metals metallic elements with atomic number greater than 20 and a density exceeding 5.6 $kg/dm^{3.}$ These metals including Lead, Cadmium, Mercury, Arsenic, and Chromium can harm both living organisms and environment posing significant health and ecological risks. They originates from human activities such farming, mining, industrial activities, and natural geology. These pollutants pose serious threat to human and the ecosystem when exposed over time. Contact with heavy metals especially at higher levels above the limits set by World Health Organization (WHO) and the

Environmental protection agency (EPA) has been increased in countries with limited water resources (Hamzat et al., 2019)

2.2.1 Sources of Heavy Metal

Heavy metals arise from various natural and human triggered sources. Some of these natural sources may be geological formation, volcanic eruption, or weathering of rocks. Heavy metal exposure may occur through multiple routes including soil, water, food, and cosmetics as these metals are naturally present in the environment (Gyamfi et al., 2023). Heavy metals enter water sources through industrial wastewater, run off and leaching from agricultural fields and industries such as mining, textiles, and battery manufacturing industries. Considerable amounts of the lead emitted from industrial process in particle size range from 0.5 to 10 µm. The uppermost levels of toxic elements such as lead are originates from these small particles (Agyarko, 2010). Waste water from textile industry, food processing industry, steel plants, coal mines, and the pharmaceutical industry contain substantial amounts of pollutants including heavy metals (Pérez, 2023). Fresh water bodies are reached by heavy metals from run-off from agricultural field introduced with pesticides and fertilizers containing heavy metals (Chahar et al., 2023). Heavy metal can enter water sources naturally through geological weathering of rocks and soil particles. Heavy metals can also reach water bodies from weathered soils/rocks (Dartey, 2015)

2.2.2 Toxicological Effects of Heavy Metals

Heavy metals comprise of essential and non-essential metals. When the essential metals are present in lower amounts, they help biological functions of living tissue and control countless biochemical processes. However, at higher concentrations heavy metals exercise a serious action on living tissues, affecting the growth, metabolism, and morphology of cells (Dartey, 2015). These metals at advanced concentrations have the capacity to cause oxidative strain by making free radicals, which can combines cellular structure proteins, enzymes and membrane systems (Apau et al., 2022).

2.2.2.1 Toxicity of Lead

Lead exists in three oxidation states: Pb (0), Pb (II), and Pb (IV), with Pb (II) being the most common form in the environment. While lead occurs naturally in the earth's crust, human activities are largely responsible for its widespread presence in the environment (Roy & Bhattacharya, 2019). Lead's extensive use in car batteries contributes to its prevalence. Prolonged exposure can lead to severe health issues, including increased blood pressure, kidney damage, brain impairment, and behavioural problems in children. Moreover, lead can cross the placental barrier, posing risks to foetal development, including brain and central nervous system damage (Roy & Bhattacharya, 2019)

2.2.2.2 Toxicity of Cd

Cadmium, a naturally occurring metal in the earth's crust, often associates with zinc. It enters the human body primarily through food consumption and accumulates in organs, particularly the kidneys (33%) and liver (14%) (Roy & Bhattacharya, 2019). Cadmium poisoning can cause severe health issues, including Itai-Itai disease, a notorious pollution-related illness in Japan. Its toxic effects include stomach pain, reproductive problems, psychological disorders, kidney damage, and immune and nervous system disruptions (Roy & Bhattacharya, 2019). Animal studies have linked long-term cadmium inhalation to increased lung cancer risk, leading the U.S. EPA to classify it as a probable human carcinogen.

2.2.2.3 Toxicity of As

Heavy metals' toxicity has been extensively documented by researchers, with arsenic being particularly notorious for its adverse effects on organisms. Arsenic exposure can lead to various health issues, including cancer, skin problems, and cardiovascular disease (H. Ibrahim, 2022). Prolonged exposure has been linked to hypertension, liver damage, and even neurological damage (Yoshida et al., 2004). Inorganic arsenic ingestion can cause distinct dermal symptoms, such as hyperkeratosis, hyperpigmentation, and hypopigmentation, as well as periorbital swelling, spontaneous abortion, and nervous system damage (Roy & Bhattacharya, 2019)

2.2.2.4 Effects of heavy metals on the ecosystem

Heavy metals pose serious risk to aquatic ecosystem. Growth, reproduction, and survival of organism are affected when the level of heavy metals in the aquatic environment increases. Extensive experience can lead to change in composition and system function of species Organisms (Dartey, 2015). They tend to accumulate when exposed over longer period since they don't, undergo biodegradation as reported by (Agyarko, 2010). Accumulated concentrations increase they move along trophic levels as it moves along the food chain. Quantities of metals increases in an organism at higher trophic level when fed on the other at lower trophic level on the food chain.

2.2.3 Regulatory Standards for Water

Countries and societies safeguard their people in drinking water to combat risk of water borne disease. Regulatory standards are set to give permissible contaminants allowed for water for human use. World health Organization, WHO, United States Environmental Protection Agency, US EPA and European Union establish standards for heavy metals. In Ghana, Ghana standards Authority set specifications drinking water. Regulatory standards for heavy metals vary depending on country and region. Table 1.1 summarizes of limits of heavy metals allowed for various metals.

Table 2.1: Regulatory standards for heavy metals in drinking water in mg/L

Heavy Metal	GS	EU	US EPA	WHO
As	0.01	0.01	0.01	0.01
Cu	2.0	2.0	1.3	2.0
Pb	0.01	0.005	0.015	0.01
Hg	0.001	0.001	0.002	0.006
Mn	0.4	-	0.05	0.4
Zn	10	-	5.0	3.0
Cd	0.003	0.005	0.005	0.003

Reference: (Arah, 2015), (GS, 2021), (US EPA, 2025), www.epa.gov, (EUR,2021).

2.3 Conventional Remediation of Heavy Metals

Researchers over time have employed various physical, chemical, biological, and advanced methods to eradicate heavy metals from water because of their persistence and toxicity. Chemical precipitation, ion exchange, membrane filtration and electrochemical treatment have been employed by earlier researchers in saving metal pollution in water. This work will dive into the various remediation methods, their advantages, and limitations to ascertain the method with higher removal efficiency and few limitations with little contamination on the environment.

2.3.1 Chemical Precipitation

Chemical precipitation, also known as coagulation precipitation. This chemical method involves addition of hydroxide (-OH) to water to form hydroxide of heavy metal. This method converts the dissolved meal ions to compact form for easy sedimentation. Conditions like PH, electro-oxidizing potential, or co-precipitation are changed. Sediment is then removed (Qasem & Mohammed, 2021). Precipitation method is relatively less expensive, simple and pH adjustment is easy. However, it is slow and produces an expensive sludge that requires further treatment. Phitany, 2025, reported that, aggregation of metal precipitates settles poorly. Improper treatment and disposal of sludge for longer time will impact negatively on the environment.

2.3.2 Ion Exchange

This is done by replacing toxic metal ion in water with less harmful metal ions like sodium or potassium using a resin. The resin is designed with functional groups that specifically target the unwanted metal ions. (Pujol, 2025) reported that, during the process, water is allowed to pass through ion exchange resin where unwanted metals are attracted to the surface of the resin and swapped with harmless ions. Ion exchange is efficient but with some drawbacks. It requires industrious cleaning of the resin to function efficiently. The cleaning involves backwash and brine regeneration. This method is not effective for higher metal ion concentrations, not decerning to heavy metals, and affected by pH of sample (Pujol, 2025).

2.3.3 Membrane Filtration

Membrane filtration employs a semipermeable membrane to remove impurities in water. Suspended solids, organic, and inorganic contaminants like heavy metals are effectively removed. Membrane filtration is categorize into microfiltration, ultrafiltration, nanofiltration, electrodialysis, reverse osmosis, forward osmosis. According to Yadav & Singh, 2021, driving forces regulating membrane process, pore size, pore distribution, degree of hydrophilicity, surface charge, solution flow, and presence of functional groups are factors that play crucial role in the interpretation of the overall membrane process of water production rate and heavy metal removal efficiency. The advantages of this method include high removal efficiency, high flux, and low energy cost, whereas the disadvantages are the high operating cost. (Yadav & Singh, 2021). Accumulation of contaminants on the membrane surface which reduce the efficiency of the membrane in removing contaminant known as fouling (Pujol, 2025).

2.3.4 Electrochemical Treatments

This electrochemical method utilizes electricity to remove heavy metal ions from water through processes like electrocoagulation, electrodeposition, electroflotation, and electrosorption. By passing an electric current through an aqueous solution with metal ions, cathode, and anode, heavy metals precipitate as hydroxides in neutral or weakly acidic conditions. According to Yadav & Singh (2021), this method efficiently removes heavy metals, producing a manageable sludge. However, its main drawback is high electrical energy consumption per contaminant mass.

2.3.5 Coagulation–Flocculation

Coagulation-flocculation is a water treatment method involving three stages: coagulation, flocculation, and separation. Coagulation destabilizes colloidal particles using coagulants like aluminum, ferrous sulfate, or ferric chloride, neutralizing their charges. Flocculation then aggregates these particles into larger flocs through gentle mixing, enhancing particle size and settleability. Finally, separation occurs via filtration, flotation, or straining (Yadav & Singh, 2021). This method is simple, cost-effective, and efficient for heavy metal removal. However, it has limitations, including potential health and environmental risks from chemical use, challenging sludge disposal, and the need for precise pH control to optimize removal efficiency.

2.4 Adsorption as an Alternative

Adsorption is a surface phenomenon that removes contaminants from water by adhering metal ions to a solid adsorbent's surface. This separation process effectively captures pollutants, allowing for their removal from water (Lupa & Cocheci, 2023). Adsorption offers several benefits, including low operating costs, high removal capacity, ease of implementation, and simple regeneration of adsorbed heavy metals (Qasem & Mohammed, 2021). Compared to other remediation methods, adsorption has advantages such as low-cost adsorbents, regenerability, no sludge formation, and potential metal recovery through desorption. Its efficiency and cost-effectiveness are particularly notable at lower metal concentrations (Yadav & Singh, 2021).

2.5 Adsorption in Water Treatment

Adsorption in water treatment involves contaminants binding to a solid phase called an adsorbent. The contaminants adhere to the adsorbent's surface, rather than penetrating its internal structure. There are two primary mechanisms: chemical adsorption, where chemical bonds form between the adsorbent and contaminants, and physical adsorption, where interactions occur without electron sharing or bond formation (Lupa & Cocheci, 2023).

2.5.1 Principles of Adsorption

Adsorption is the selective concentration of components from a mixture onto a solid surface. The binding process is influenced by factors like adsorption type, kinetics, isotherms, and surface energy. Adsorption can be classified based on the strength of interactions between the adsorbent and adsorbate. Physical adsorption involves weak forces (Vander Waal's and electrostatic) and is reversible. Chemical adsorption, on the other hand, forms strong chemical bonds, altering electronic structures and bond properties, making it irreversible(Ranke, 2008).

2.5.1.1 Adsorption isotherm models

The adsorption isotherm illustrates the relationship between the equilibrium concentrations of adsorbate in solution and the amount of adsorbate on the adsorbent, describing the distribution of adsorbate molecules between the liquid and solid phases at equilibrium (Dehmolaei & Vadi, 2014). Common isotherm models include Langmuir, Freundlich, Temkin, Dubinin-Raduskevich (D-R), and Brunauer-Emmett-Teller (BET). This study utilized the Freundlich and Langmuir isotherms to understand the adsorption of heavy metals onto ACNT, optimizing conditions and designing efficient systems.

2.5.1.2 Langmuir Isotherm

The Langmuir isotherm model assumes monolayer adsorption onto a homogeneous surface with a finite number of specific sites, where each site adsorbs one molecule. Once a site is occupied, no further adsorption occurs (Dehmolaei & Vadi, 2014). The Langmuir constant (KL) can predict the affinity and favorability between the adsorbent and adsorbate through the dimensionless separation factor (H. Ibrahim, 2022). This model is based on a simple physical adsorption mechanism, assuming a uniform surface with discrete active sites and no interactions between adsorbed molecules

2.5.1.3 Freundlich Isotherm

Freundlich isotherm describes adsorption on heterogenous surface with non-uniformly distributed adsorption sites**.** This empirical model assumes that the amount adsorbate at equilibrium is related to the adsorbent concentration through a power law relationship. The Freundlich isotherm model is expressed as

$$\log q_e = log k_f + \frac{1}{n}\log C_e \qquad \text{Equation (1)}$$

K_f represents the adsorption capacity

n- adsorption driving force

q_e – amount of adsorbate

Ce – equilibrium concentration of adsorbate

It has been established that as the K f increases, the adsorption capacity increases including adsorption strength (n). Generally, for good adsorption, the estimated values of n ranged between 2–10 with 1–2 for moderate and <1 for poor adsorption (Hamzat et al., 2019).

2.5.1.4 Adsorption kinetics

In designing and optimizing adsorption system, understanding adsorption kinetics cannot be overlooked. Adsorption kinetics describes the rate at which molecules in an adsorbate bind to adsorbent. Kinetic models help in predicting adsorption performance of an adsorbent, optimizing operation conditions, and designing efficient adsorption system

Adsorption kinetics models describe the rate of adsorption. Pseudo-first-order assumes adsorption rate depends on available sites, while pseudo-second-order assumes it's proportional to the square of available sites. Elovich model describes heterogeneous adsorption with decreasing rate as surface coverage increases. Bangham model attributes adsorption rate to pore diffusion. In a study, pseudo-second-order kinetics best described Cr and Zn adsorption onto P-CNTs, while PEG-CNTs followed the order: pseudo-second-order > Elovich > pseudo-first-order (Hamzat et al., 2019).

2.5.2 Factors Affecting Adsorption Efficacy

Some features that impact on rate of adsorption include temperature, pH, Surface area of adsorbent, adsorbate concentration.

2.5.2.1 Consequence of pH

pH affects adsorption due to hydrogen ions that occupy adsorbent surface. According to (Liu et al., 2012), maximum adsorption recorded ranges from slightly acidic medium to the neutral due to reduction of hydrogen ions in the sample. In this study pH was kept around the neutral range to reduce the concentration of hydron ions in the system and to keep the drinking water pH.

2.5.2.2 Effect of contact time

Increase in interaction period upsurges the adsorption (Baby & Saifullah, 2019). This result from increase in the interaction of between the adsorbate and the adsorbent. ***(***Hamzat ***et*** al***.,*** 2019***)*** observed that Cr and Zn ions are greatly removed increases with increasing contact time notwithstanding of the nano-adsorbents until an optimum time of 20 min for different nano-adsorbent

2.5.2.3 Effect of quantity of adsorbent

The effects of different mass of nano-adsorbents dosage (0.1–0.5 g) on the certain heavy metals elimination from battery wastewater were investigated. Results depicted that the percentage of elimination increases with increasing quantities of adsorbent material (0.4 g for P-CNTs and 0.3 g for PEG- CNTs), (Hamzat et al., 2019). The increasing adsorption efficiency was attributed to large surface area availability and the existence of functional binding sites.

2.5.2.4 Effect of temperature

Temperature plays significant role in adsorption of heavy metals onto suitable adsorbents. Increase in temperature increases the energy in the system which significantly increase the interaction of adsorbent with adsorbate. A study conducted by Hamzat et al., 2019, depicted that increase in temperature from 303 to 343K was responsible for the corresponding increase in the removal efficiency and adsorption capacity of the selected heavy metal ions onto P- CNTs and PEG-CNTs. The detected phenomenon was ascribed that an increase in temperature triggered a swelling effect in internal and external edifice of the nano-adsorbent. This invariably led metal ions to penetrate the active sites of nano-adsorbent increasing adsorption efficacy. P-CNTs and PEG-CNTs recorded maximum adsorption of metal ion at 333K and 323 K, respectively

2.6 Carbon-Based Adsorbents

Carbon-Based adsorbents have been widely explored and applied in areas of science for their exceptional high surface area, chemical stability, and versatility. In water treatment, carbon-based adsorbents such as activated carbon, graphene and carbon nanotube are used in removal of heavy metals and organic waste. Lupa & Cocheci, 2023, conducted a study on diverse carbon-based adsorbents in eliminating different impurities in wastewater using different methodologies. The results depicted an increase in adsorption capacities from porous activated charcoal to functionalized carbon nanotubes with the highest

2.6.1 Activated Charcoal

Activated carbon is carbon material produced from organic material specially treated through pyrolysis and activation to increase its surface area and adsorption capacity. It may be produced from wood, coconut shells and coal. Activated carbons are industrially produced carbonaceous products with spongy structure and a large inner surface area (Scholz, 2023). Particle size ranges from 3mm and smaller pore volume of 0.2 mL/g. Internal area is greater than 400 m^2 /g. The opening size ranges from 0.3 to a few thousand nanometers as said by (Duruaku, 2012). The pore structure created in the activated carbon and its characteristics depend on the untreated material and the engineering process. The basic chemical structure of activated carbon and pure graphite are closely related. The graphite crystal consists of layers of fused hexagons held together by weak van der Waals forces (Scholz, 2023). The structure of activated carbon differs from that of graphite in terms of the distance between layers. The average distance between layers in activated carbon ranges from 0.34 to 0.35 nm , while 0.33 nm in graphite (Ganjoo et al., 2023).

AC is used in treating poisoning and overdose of drugs, air sanitization, hydrogen storage, decaffeination, air purification, capacitive deionization, gold extraction, solvent recovery, super capacitive swing adsorption, metal extraction, and sewage treatment (Rakhanawati et al, 2020). In water treatment, AC is used as a bacteria inhibitor, heavy metals removal and other pollutants. Studies show that AC has high removal efficiency of heavy metals between 0.08 mml/g to 0.175 mol/g as reported by (Lupa & Cocheci, 2023). AC as adsorbent has a limitation like other adsorbents including cost and regeneration. AC may be costly when not synthesized from organic waste sources like coconuts husk and wood shavings.

AC regeneration involves eradicating adsorbed materials from AC by physical or chemical techniques without fluctuating its inventive structure and reinstating its adsorption efficiency for reprocess. There are many regeneration methods including thermal regeneration, biological regeneration, wet oxidation regeneration, electrochemical regeneration, solvent regeneration, and catalytic wet oxidation (Ganjoo et al., 2023). Desorption is carried out by reducing the pressure or increasing the temperature. However, complete desorption of a substance from the inner surface is only possible with difficulty, since the heat of desorption is greatest with small loads. the problem can arise that the desorption temperature rises above the decomposition temperature of the compound in the process. A residual load consisting of pyrolysis products and carbon deposits remains in the active centers after desorption, which reduces the adsorption capacity of the carbon due to temperature, (Scholz, 2023). Regeneration requires expertise and machinery which is cost involving.

2.6.2 Carbon Nanomaterials

Carbon-based adsorbents like activated carbons (ACs), carbon nanotubes (CNTs), fullerenes, and graphene (GN) are widely used for heavy metal removal due to their large surface area (500-1500 m^2/g). Carbon nanomaterials can be classified based on their dimensions: zero-dimensional (0-D, e.g., fullerenes, quantum dots), one-dimensional (1-D, e.g., carbon nanotubes), two-dimensional (2-D, e.g., graphene), and three-dimensional (3-D, e.g., graphite) materials(Baby & Saifullah, 2019).

2.7 Carbon Nanotubes (CNTs)

Carbon nanotubes (CNTs) are cylindrical nanostructures composed of sp2 hybridized carbon atoms arranged in a hexagonal lattice. Their unique tubular structure and high aspect ratio confer distinct properties, making them valuable in material science for various applications (S. O. Ibrahim et al., 2020.).

2.7.1 Structure and Properties of CNTs

Carbon nanotubes (CNTs) have undergone significant research advancements over the past three decades, showcasing exceptional mechanical, electrical, and thermal properties. These properties make them ideal for applications in electric devices, sensors, and energy harvesting systems (Nguyen et al., 2024). CNTs are classified into single-walled (SWCNTs) and multi-walled (MWCNTs) types, based on the number of graphene layers. SWCNTs have diameters ranging from 0.7 to 1.2 nm, while MWCNTs have diameters between 4-30 nm. Both types exhibit similar thermal and electrical conductivities. MWCNTs have been widely used as adsorbents for removing pollutants from water due to their large surface area, high aspect ratio, and unique structure (Hamzat et al., 2019). Their properties depend on atomic arrangement, diameter, length, and morphology (Hamzat et al., 2019).

2.7.2 Adsorption Mechanisms of CNTs

Carbon nanotubes (CNTs) have attracted significant research attention due to their unique structural and chemical properties, enabling effective adsorption of pollutants from water. The adsorption mechanism plays a crucial role in optimizing CNT design for water treatment. Key mechanisms include physical adsorption via weak van der Waals forces, chemisorption through chemical bonding, and electrostatic forces (Saleh et al., 2024). Temperature, pH, adsorbate quantity, surface area, and functional groups significantly impact these mechanisms (Nyairo et al., 2022). Functional groups like -COOH, -NH2, and -OH enhance adsorption capacities through complexation, hydrogen bonding, and coordinate bonding (Yi et al., 2023). Additionally, π-π interactions between CNTs and aromatic rings facilitate adsorption of aromatic compounds (Li et al., 2022).

2.3.6 Limitations of Pristine CNTs

Pure carbon nanotubes (Pristine CNTs) that are free from impurities, functional group and surface defects have perfect hexagonal lattice structure and exhibit exceptional mechanical, electrical and thermal properties. It is used as reference for studying functionalization and surface defects of carbon nanotubes but exhibit limitations such as cost, limited surface-active sites, hydrophobicity, and aggregation in aqueous solutions.

Pristine CNTs require special synthesis methods with control over conditions such as higher temperature, use of high purity percussors and minimize contamination which affects the cost of production. Pristine CNTs repel water due to non-polar graphitic surface affect dispersion in aqueous solution. This affect its ability to be used in water treatment. Pristine CNTs bunch to minimize exposure to water caused by strong forces between particles (Pérez, 2023).

To overcome these limitations, activation and functionalization of CNTs is done to improve surface sites and water solubility for water treatment application.

2.7.4 Carbon nanotubes in Water Treatment

Clean water is an essential resource for human life and our ecosystem. However, due to the increasing growth of population and rapid development of economy and urbanization, the increasing pressure on water scarcity issue highly requires cost-effective water treatment technologies to produce high-quality clean water (Ma et al., 2017).

Different treatment techniques used to remove heavy metals from water include reduction and precipitation, coagulation and flocculation, ion exchange, membrane technologies, electrolysis, ozonation reverse osmosis, electrochemical separation, and electro coagulation (Hamzat et al., 2019). Despite the satisfactory results obtained via these technologies, there exist some limitations such as large capital or operational costs of chemicals reagents, generation of toxic sludge, and ineffectiveness removal of heavy metals (Hamzat et al., 2019).

Traditional water treatment techniques, including filtration, distillation, coagulation–flocculation, bio sand and reverse osmosis fail to remove all heavy metal ions from the water sample. Adsorption technology is considered more helpful compared to other techniques due to its simplicity, relative availability, low cost and effectiveness in the removal of potential toxic element from very dilute complex solutions containing diverse pollutants (Hamzat et al., 2019).

At present, carbon nanotubes have received widespread attention in the field of water treatment and have been extensively studied in desalination, removal of heavy metal ions, and oil-water separation (Li et al., 2022).

According to Baby & Saifullah, 2019 carbon nanotubes are promising advanced materials for the catalysis because of their improved quantum efficiency, nano-size, high chemical stability, hollow tube structure, and extended light adsorption region due to their large specific surface area (Mauter &Elimelech 2008). An investigation was designed on photocatalyst-based SWCNTs-TiO2 and results showed successful application for the purification of water from oil (Gupta et al 2017)

2.8 Activated Carbon Nanotubes (ACNTs)

2.8.1. Synthesis Approaches

Carbon nanotubes are typically synthesized using three main methods: arc discharge, laser ablation, and chemical vapor deposition (CVD). CVD involves a chemical reaction in a vapor phase, forming a solid product on a substrate surface (Hussain, 2014). However, this method often results in impurities and requires complex equipment. Arc discharge produces nanotubes through carbon vapor, while laser ablation yields high-purity SWNTs. This study employed the modified Staudenmaier method, a simple and instrument-free approach, to synthesize multi-walled carbon nanotubes (Saleh et al., 2024).

2.8.2 Activation of Carbon nanotubes

Activation is the process of modifying the surface properties with the aim of increasing reactivity and interaction with other materials. The various method used include chemical activation, physical activation, and bio-mass derived CNT activation.

Chemical activation involves treatment of CNTs with strong acids, oxidizing agents or amines or alcohol with the aim of modifying their surface properties (Nyairo et al., 2022). This is used to introduce functional group, improved dispersion in solvents and enhance interaction with other materials (Saleh et al., 2024). The limitation is that damage to structure and introduction of impurity if not carefully done. Carbon nanotubes can be exposed to plasma, exposure to UV light, higher temperatures, and grinding to improve mechanical, electrical, and heat conductivity. Again, surface defects may be caused, and modification may not be uniform and high cost involved due to specialized equipment needed. Biomass-based activation employs hydrothermal treatment, biomass-based physical activation, or biomass-based chemical activation. These methods can be cost effective, ecofriendly and can be used to adjust specific features of CNTs for precise uses but may contain impurities and can be challenging due to variability of biomass source.

2.8.3 Characterization of ACNTs

The properties of ACNTs and MCNTs were investigated using various characterization techniques, including field emission scanning electron microscopy (FESEM) with an EDX detector and X-ray diffraction (XRD) analysis. The study utilized advanced instrumentation, such as ZEISS Supra 40VP and Bruker Advance D8, to examine the physical properties of ACNTs. Additionally, techniques like SEM, FTIR, UV-Vis spectroscopy, and XRD were employed to determine the properties of synthesized ACNTs, providing a comprehensive understanding of their structure and characteristics. These techniques enable researchers to analyze the morphology, composition, and crystal structure of ACNTs, which is crucial for understanding their properties and potential applications (Saleh et al., 2024)

2.8.4 FTIR Analysis of ACNTs.

FTIR analysis reveals the surface modification, chemical structure, functional groups, and impurities in ACNTs. Functionalizing carbon nanotubes with specific groups enhances their properties, dispersion, reactivity, and interactions with other materials (Smith, 2018). FTIR spectra can identify characteristic bands, such as aromatic rings (C=C bond) at 1600 cm-1 and hydroxyl groups, which play a crucial role in adsorption (Wang et al., 2020). The presence of peaks between 3800-3200 cm-1 may indicate moisture absorption or purification processes. Other peaks, like C-H stretching methylene (CH2) at 2910-2940 cm-1 and asymmetric CH2 bending at 1450 cm-1, provide insights into the CNTs' structure and defects.

2.8.5 SEM analysis of ACNTs

Electron microscopy is crucial for characterizing CNTs, allowing direct observation of size, shape, and structure. Techniques like TEM and SEM are commonly used to examine exfoliation, purity, and morphology. SEM provides information on surface morphology, chemical composition, and diameter of CNT bundles, while TEM directly measures nanotube diameter. SEM is particularly useful for analyzing CNTs and their composites, revealing fibrous bundles and any changes in tubular structure. In this study, SEM was used to perform morphological analysis, examining diameter, length, and aggregation state (Sonkar & Kumar, 2022). Any small change in the tubular structure can be identified using this technique. (Aslam et al., 2021) used scanning electron microscopy (SEM) evaluates the nanostructure of the tubes

2.8.6 XRD analysis.

XRD is the most confident method for crystal characterization though it also has its own limitations. The monochromatic beam is obtained from polychromatic beams produced in a special tube called cathode ray tube, the monochromatic rays then hit on to the sample atomic planes. (Itas et al., 2020). XRD has successfully been utilized to enlighten the morphology and structural features of carbon nanotubes aligned at different angles. The carbon atoms in carbon nanotubes act as 3D optical diffractors that scatter light at different, but specific angles. From the diffracted angles, it would be possible to extract information on aligning graphene sheets of carbon nanotubes from the position and intensity of diffracted beams (Itas et al., 2020).

2.8.7 UV VIS analysis

UV-Vis spectroscopy is a powerful tool for studying the optical properties of carbon nanotubes, providing insights into their absorption and scattering behavior. The absorption spectra reveal information about their electronic structure, including van Hove singularities (Liu et al., 2018). This technique enables real-time monitoring of CNT dispersion in aqueous media, crucial for producing consistent nanocomposite products. Extensive research has demonstrated the effectiveness of UV-Vis spectroscopy in monitoring CNT dispersion quality UV-Vis spectroscopy is a powerful tool for studying the optical properties of carbon nanotubes, providing insights into their absorption and scattering behavior. The absorption spectra reveal information about their electronic structure, including van Hove singularities (Liu et al., 2018). This technique enables real-time monitoring of CNT dispersion in aqueous media, crucial for producing consistent nanocomposite products. Extensive research has demonstrated the effectiveness of UV-Vis spectroscopy in monitoring CNT dispersion quality (Njuguna et al., 2015).

2.9 Adsorption of Heavy Metals Using ACNTs

Heavy metal pollution poses significant environmental and health risks, prompting research into efficient removal technologies. Adsorption using activated carbon nanotubes (ACNTs) shows promise due to its high surface area, chemical stability, and targeted applications. ACNTs effectively remove a wide range of heavy metals, including lead, mercury, cadmium, and arsenic, making them ideal for water purification and environmental remediation (Nyairo et al., 2022).

2.9.1: Adsorption of As, Cd, Cr, Hg and Pb

Table 2.2: Adsorption of heavy metals by activated carbon nanotubes

METAL	ADSORPTION CAPACITY	MECHANISM	MODEL	REERENCES
Pb^{2+}	ACNTs have shown high adsorption of Pb with maximum adsorption of 15.9 mg/L at a pH of 5.3 with initial concentration of 30mg/L	The adsorption was attributed to Electrostatic, hydrophobic, and π–π interactions interaction between ACNTs and the metal ions	Langmuir isotherm	(Aslam et al., 2021)
Cd^{3+}	ACNTs exhibited maximum adsorption of 169.2mg/L at pH of 6 and 298K	Adsorption was due to electrostatic interaction, physical adsorption, surface precipitation	Langmuir isotherm	(Nyairo et al., 2022)
As^{3+}	ACNTs showed adsorption efficiency of 87.6% at pH of 2.6	Adsorption occurred because of Liquid film diffusion, ion exchange	Tempkin, Dubinin-Radushkevic, Langmuir, Freundlich	(Baby & Saifullah, 2019)
Cu^{2+}	ACNTs removed a maximum of 16.95 mg/L at pH of 5 and temperature of 298K	Adsorption took effect by chemical interaction	Langmuir	(Nyairo et al., 2022)
Hg^{2+}	ACNTs adsorbed 72.5% at pH of 6	Adsorption was done through electrostatic interaction	Langmuir	(Mallakpour & Khadem, 2019)

2.10 Regeneration and Reusability

Regeneration is the process of removing pollutants from an adsorbent restoring its adsorption capacities and making it effective for reuse. ACNTs just like other carbon-based adsorbents can be regenerated through several methods. An investigation by (Mallakpour & Khadem, 2019) revealed that after adsorption process, eliminating the solid contaminants from aqueous media was a thoughtful problem in environmental remedy. Centrifugation and filtration methods can separate adsorbents from solution. Extra stage and costs required this treatment. In this regard, the magnetic-separation technology was projected as auspicious ways to deal with such problem. Exclusion of solid particles based on their magnetic performances has received the attention of many scholars. According to (Baby & Saifullah, 2019) ACNTs is easy to regenerate and reused making it suitable for removal of contaminants and heavy metal treatment in water. Regeneration is also known as desorption.

2.10.1 Desorption techniques

When an adsorbent is saturated with adsorbent or nearly exhausted, adsorption is stopped adsorbent is subject to desorption. The following methods may be employed in desorption process. Water washing desorption is also known as backwash (EUWA, 2018). This method employs use of water flow to remove pollutants from activated carbon nanotubes. This method is suitable for water soluble pollutants. It is ecofriendly, easy to operate, and low energy is required. This method may not be feasible for non-water-soluble pollutants and high concentration of heavy metals.

Thermal regeneration involves high temperature heating to break down adsorbed pollutants. Desorption is done by increasing the temperature of the adsorbent by passing a hot carrier or purge or stripping gas like high temperature steam over it or by dropping the pressure in the adsorbent bed. Marques et al., 2017 presented that it is done by applying heat to the carbon containing adsorbed pollutants under passive atmosphere, to degrade the adsorbate and recuperate the largest possible part of the permeability. It is suitable for organics pollutants from industrial waste and wastewater treatment. High energy require is the limitation for this technique.

Chemical regeneration uses specific chemical solvent to remove adsorbed pollutants from adsorbent. This method is good for adsorbed heavy metals and insoluble organic matter. Specialized equipment technical expertise required is the limitation for this method.

Biological regeneration is the microbial decomposition of organic pollutants adsorbed on adsorbent. Lower energy is required by this method, suitable for long term operation in wastewater treatment but method is very slow.

2.10.2 Cyclic stability of ACNTs.

Cyclic stability of ACNTs is the ability of carbon nanotubes to maintain structure adsorption abilities over repeated cycle of used. Studies has sown varying degree of stability of ACNTs depending on type of adsorbate removed, regeneration method used and the structure of the ACNTs.

Cyclic stability of ACNTs is important in environmental remediation since it can help reduce the amount of waste generated from used ACNTs. Reduction in the regular need for replacement and regeneration of ACNTs can lead to lower environmental footprint.

Cyclic ability of carbon nanotubes is crucial since it can lead to significant cost saving by reducing resources required for production and regular replacement of ACNTs. The efficiency is maintained for longer period of use and reuse of ACNTs.

2.11 Research Gaps

Despite the progress made on synthesis, characterization, and application of ACNTs in scientific investigations due to their promising impact on potential application and environmental remediation, there are several research gaps

- Limited studies on biomass-derived ACNTs for heavy metal removal which is cost effective for large scale production and industrial application.
- Lack of comparative analyses with conventional adsorbents.
- Insufficient data on adsorption mechanisms and kinetics for certain metals.
- Need for large scale and real wastewater application studies.

2.12 Conceptual Framework

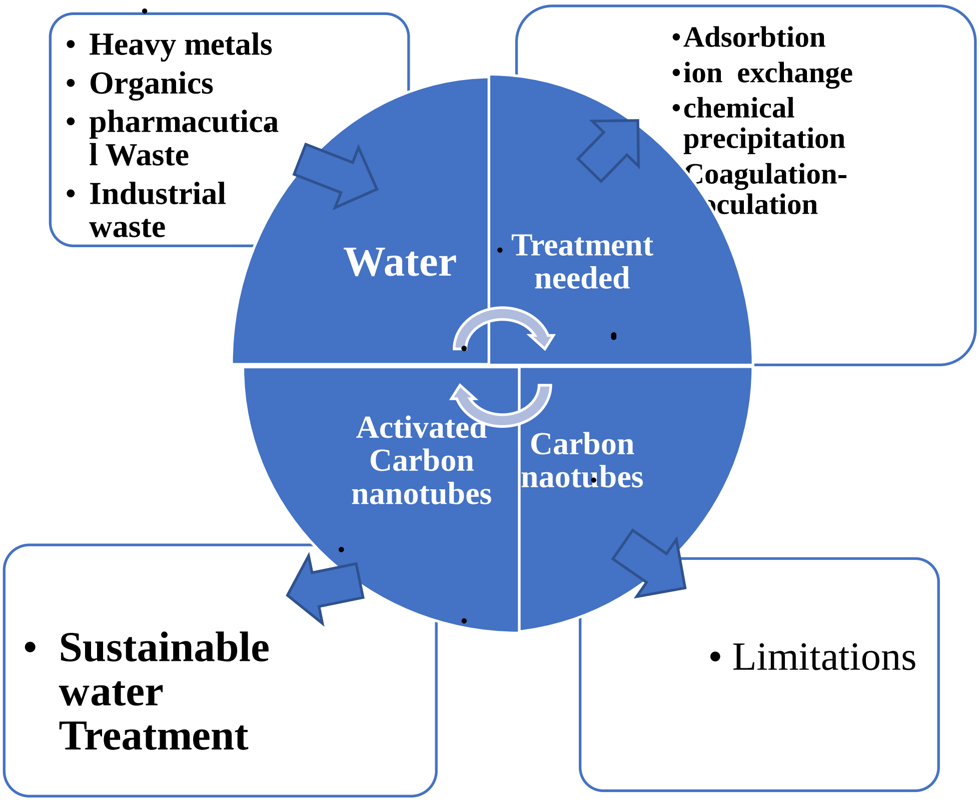

Figure 2.1: Conceptual framework of water treatment by adsorption with ACNTs

2.13 Summary of the Literature Review

In this section, detailed review of existing literature on synthesis, characterization and evaluation of ACNTs in removal of heavy metals. This educative review highlights the ability of ACNTs as effective adsorbent for water purification including heavy metals, organic toxins, and other contaminants.

Synthesis methods are detailed discussed pointing out their strengths and weaknesses of each. Synthesis from biomass percussors, its advantages and limitation are also examined. Activation techniques including chemical activation and physical activation are discussed. Characterization techniques to evaluate surface area, pore size, functional group, morphology, and crystallinity are reviewed.

Application of ACNTs in heavy metals treatment reveals their excellent adsorption efficiency, selectivity reusability and cyclic ability compared to another carbon-based adsorbent. Challenges and limitations of ACNTs such as cost and potential environmental impact are also discussed.

This review has provided insight on several research gaps. This work will address the existing knowledge on synthesis of ACNTs from low cost percussor like graphite powder. The investigation will also provide in-dept knowledge on adsorption mechanism of metals like

Pb, As and Cd. By building on existing knowledge, this research intends to contribute to sustainable and effective water treatment technologies. In short, this review provides thorough understanding of the current state of research on ACNTs in treatment of impurities in water and identifies areas for future investigations and development.

CHAPTER THREE

METHODOLOGY

3.1 Introduction

This episode presents the experimental approaches and methods used in the synthesis, characterization, and application of Activated carbon nanotubes in eliminating selected heavy metals in water from hand-dug wells. The methodology delineates detailed account of materials, instruments and protocols utilized in the study allowing replication and validation of the results.

The materials, equipment and procedures employed in the synthesis of carbon nanotubes, instrumental analysis for characterizing the synthesized carbon nanotubes. Adsorption studies, sampling, application of synthesized carbon nanotubes on water samples, analytical methods employed in determining the target impurities in water samples before and after application and methods for analyzing data is clearly outlined. By detailing the procedures and techniques, this chapter facilitates duplication and informs the analysis of results presented later.

3.2 Study Area

The study was conducted in Berekum, a municipality in Ghana's Bono region. Berekum is bounded by Tain and Jaman North to the north, Dormaa East to the south, Dormaa Municipal and Jaman South to the southwest and west, and Sunyani West to the east. The area features a tropical savanna ecosystem with two forest reserves: Tain and Pamu-Berekum. Its wet semi-equatorial climate has two rainy seasons, making it suitable for diverse crops and livestock production. The population engages in various economic activities, including industry, agriculture, and trade. However, rapid population growth has led to inadequate waste disposal facilities. The main sources of drinking water are Mechanized wells/pipe-borne water 38%, Streams/Rivers 31%, Hand-dug wells 19%, Boreholes 10%, Bagged/Mineral water 2%. This indicates about 69% of the population has access to potable drinking water (MOFA, 2023).

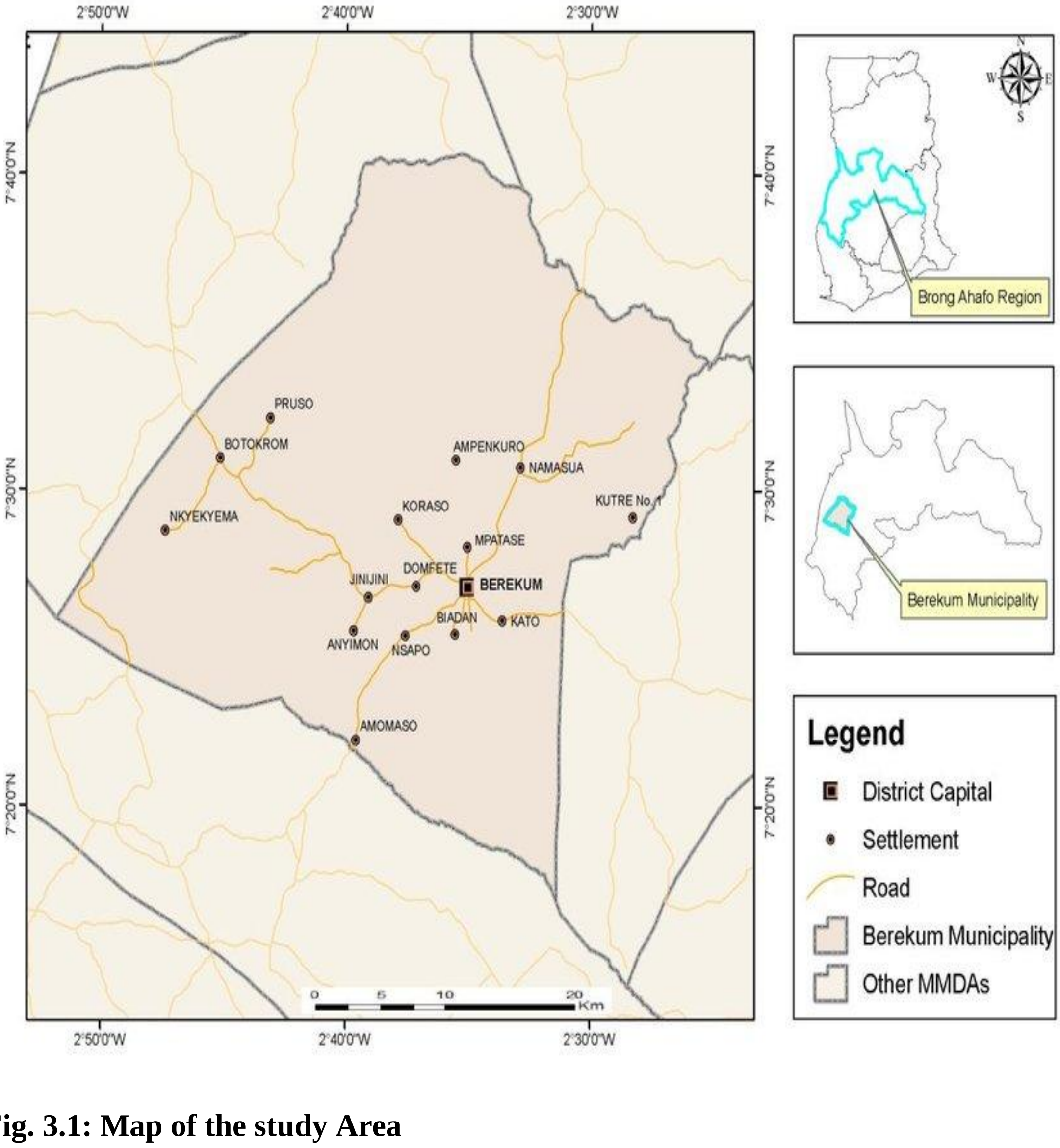

Fig. 3.1: Map of the study Area

3. 3 Synthesis of Carbon Nanotubes

3.3.1 Chemicals and equipment

3.3.1.1 Chemicals

All chemicals were of analytical of analytical grades manufactured by KANTO CHEMICAL CO, INC, Japan. Chemicals were purchased from LAB COMPANION LIMITED, Accra, Ghana. The chemicals include.

- Graphite powder (Sigma-Aldrich 99.99% purity)
- Conc. nitric acid (HNO_3 69%)
- Conc. Sulfuric acid (H_2SO_4 98%)
- Potassium Chlorate ($KClO_3$ 99.5%)

3.3.1.2 Equipment

- Ice bath (Clifton model 88392, Nickel-Electro Ltd, England) 5 °C
- Sand bath (China) 70°C
- Glass funnel (100mm Diameter)
- Oven (Wagtech model GP/50/CLAG250/HYD, Wagtech Projects, England) at 70°C
- Chemical balance (Kern Model PCB 1000-2, KERN & Sohn GmbH, Germany) with 1000 $\pm$0.01
- Magnetic stirrer (Stwart model SP161-3, Bibby Scientific, England) 6.5 rpm

3.3.2 Cleaning of Glassware

Glassware was soaked for 24hours to remove impurities in glassware that may taint the preparation in chromic acid. 5 g of solid K_2CrO_7 salt was dissolved in 100 mL 0.1 M H_2SO_4. Glassware was further washed with detergent to remove further chemicals and later washed with distilled water to make it free from impurities that may affect the synthesized carbon material. Glassware was dried for 24 hours in 70˚C oven to remove water stains.

3.3.3 Synthesis of Carbon nanotubes from Graphite powder

10g of graphite powder was weighed and transferred into a mixture of fuming 50 mL HNO_3 and 100 mL H_2SO_4 slowly while stirring at 6.5 rpm for 1 hour to disperse carbon particles and to prevent aggregation of carbon particles.

Mixture was cooled on an ice bath to 5 ˚C. 50 g of $KClO_3$ was slowly added to breakdown graphite particles down to smaller particles for 1 hour. $KClO_3$ was added on an ice bath because the reaction is highly exothermic and require very low temperature to prevent the reacting vessel from breaking and splashing out of concentrated acid from the reaction vessel. The resulting solution was heated on sand bath up to 70 ◦C for 72 hours for carbon particles in the mixture to foam throughout the mixture. The resulting mixture was then open in air for 72 hours for carbon particles to settle based on their sizes. The floating carbon material were carefully filtered and was stirred for 2 hours in 1000 mL deionized water. Carbon particles were then filtered from the solution and dried for 4 days at 30 ◦C (Saleh et al., 2024), (Lee and Seo, 2010).

3.3.4 Activation of Activated carbon nanotubes

0.1 g of synthesized Activated carbon nanotubes was added to a mixture of 4.0 M nitric acid and 10.0 M sulfuric acid while stirring at a speed of 6.0 rpm. 5mL of nitric acid was mixed with 15mL sulfuric acid to prevent damage and stirred for 12 hrs.

The mixture was then washed in 1000 mL deionized until neutral pH was reached. Carbon nanotubes was filtered and dried at room temperature (Saleh et al., 2024). Synthesized carbon material was sent to Laboratory and Regional Water and Environmental Sanitation Center (RWESCK), at Kwame Nkrumah University of Science and Technology (KNUST), Ghana for instrumental analysis.

3.4 Characterization of Activated Carbon nanotubes

FTIR, SEM, UV-Vis and XRD analysis were performed to determine surface morphology, functional group and crystallinity of synthesized activated carbon nanotubes.

3.4.1 FTIR Analysis of activated Carbon nanotubes

FTIR analysis was done to establish the molecular structure of carbon using Bruker FTIR instrument from United States of America with OPUS version 7.2.13 9.1294 with scan range of 400-4000 cm^{-1} and transmittance range of 0-70%. The advanced features and recent software of the FTIR helped to ascertain chemical modifications of the activated carbon nanotubes and presence of impurities

3.4.2 UV–Vis analysis of activated carbon nanotubes

UV analysis of carbon nanotubes was done at the using UV-Vis Spectrometer model 223E1451 (Analytik Jena GmbH, Germany). The spectral scan range between 200-800nm and a speed of 50nm/s to determine the π-plasmon adsorption and concentration of activated carbon nanotubes (Kataura et al., 2009) and (Liu et al., 2011). W-lamp was switched at 320 nm to ensure optimal performance and accuracy across the range. The spectral scan mode and high-resolution settings enables detailed absorbance which can offer understanding into the electronic structure and chirality of the activated carbon nanotubes.

3.4.3 XRD analysis of activated carbon nanotubes

X-Ray examination was done with X'Pert3 Powder PANalytical high precision X-ray Diffraction instrument from England at Ka radiation (λ=1.5406) na to determine structural properties such as crystallinity, tube diameter, structural order, chirality, and defects. Three different angles of incidences were used to ensure that crystal structures and lattice parameter were accurately determined.

3.4.4 SEM analysis of Carbon nanotubes

Microscopic analysis was performed on synthesized Activated carbon nanotubes to investigate the surface properties and structure using Zeiss Sigma SEM from Germany with 5 kV accelerating voltage, 9.18 mm working distance and magnification of 1.77 KX to provide comprehensive view of the sample. The sputter coater was then used to apply a thin 10 nm conductive coating to prevent charging and improves imaging. Sputter coated sample was inserted into SEM chamber and image under vacuum at 15 kV accelerating voltage. The instrument was operated in different magnification ranges to detailed surface morphology of sample

3.5 Adsorption studies on Activated carbon nanotubes

Maximum adsorption conditions were investigated using Batch adsorption varying (0.1 g, 0.2 g, 0.3 g, 0.4 g to 0.5 g) of Activated carbon nanotubes varying contact time (10, 20, 30, 40, 50, 60 minutes) were investigated using 0.01 mL^{-1} solution of each metal at 6.5 rpm. 50 mL of the solution of the standard metal was used in each experiment.

Optimum conditions determined were based on the maximum adsorption capacity (Kumar et al., 2019).

3.6 Sampling

Three wells, one from each community is selected at random and water sample is taken from each well.

3.6.1 Sampling process

A 3 L plastic bucket with a 50 m rope attached to its handle was used. The bucket was lowered deep into the well until 5 m of the rope removed. The bucket was filled with water and was poured back to ensure the content of the wells were uniformly mixed before samples were taken into a 1 L plastic container. The 1L plastic container was filled to its brim and corked with its lid and labelled. This sampling procedure was repeated in each sampling point in all the three communities where the wells were located.

3.6.2 Handling, Storage and Preparation of Samples

Water samples were collected with samples bottles cleaned with soap and rinsed three times with water samples before 500 mL of samples are taken from individual sample site. Each sample will be identified with label consisting of community name. Samples were transported to Koase SHS lab for digestion for heavy metal determination and treatment with synthesized activated carbon nanotubes.

3.7 Preparation of samples for Pb, As, and Cd analyses.

For heavy metal analysis, acidified water samples underwent digestion following this procedure.

100 mL of acidified water sample was mixed with 5 ml concentrated HNO_3 and 5 mL concentrated H_2SO_4.The mixture was heated to reduce the volume to 15-20 mL, allowing acid concentration. The digested sample cooled to room temperature. It was filtered through 0.45 μm Whitman filter paper. The final volume was adjusted to 100 mL with double distilled water and stored for analysis (APHA, 2017). This method was applied to all samples at Koase STEM/SHS Chemistry Lab in Wenchi.

3.8 Determination of concentration of Pb, As and Cd in raw water

Digested water samples were sent to Council for Scientific and Industrial Research (CSIR)-Forestry Research Institute at Fumasua, Kumasi for analysis using Atomic Absorption Spectroscopy (AAS). Samples were analyzed three times and the mean concentrations were recorded and presented.

3.9 Treatment of water samples with Activated carbon nanotubes

The raw water samples were Activated carbon nanotubes were centrifuged at 320 rnp for 2 minutes was transferred into a 250 mL Pyrex flat bottomed flask and digested using the digestion process outlined for the raw water samples. The samples were analyzed in triplicates using AAS. The mean concentrations were recorded and presented

The efficiency of the synthesized activated carbon nanotubes was determined and expressed as a percentage using: Efficiency, $E = \frac{C_0}{C_e} \times 100\%$

Equation (2)

Co – concentration of metal removed

Ce – initial concentration of metal present in sample

3.10 Analyses of results

Data from characterization, and application of activated carbon nanotubes for groundwater treatment were interpreted and analyzed using various statistical and computational methods. The following steps were taken:

3.10.1 AAS Results of metal ion concentration

The collected data was organized using Microsoft Excel to ensure accuracy and consistency. The efficiency of the synthesized carbon nanotubes was determined and expressed as a percentage using:

$$\text{Efficiency, E} = \frac{C_o}{C_e} \times 100\% \qquad \text{Equation} \quad (3)$$

Where C_o is amount of metal removed,

C_e is the initial amount of metal in sample

Mean, median standard deviation and coefficient of determination were performed with SPSS to identify trends and correlations in the data.

3.10.2 Results on characterization

Instrumental data from FTIR, SEM, XRD and UV-Vis were analyzed using computer software applications. SEM images were analyzed with Image J to interpret the size of particles, shape, and diameter as well as surface features. FTIR results was interpreted using OriginPro application to determine the peak positions and intensities.

3.10.3 Adsorption Isotherms

Nonlinear regression analysis was used to fit adsorption isotherm models to experimental data, elucidating the adsorption mechanism. Both Langmuir and Freundlich isotherm models were applied to determine the best fit and understand contaminant removal by carbon nanotubes. The Langmuir model, suitable for monolayer adsorption, and the Freundlich model were evaluated for their ability to describe the adsorption behavior is shown in the following equation:

$$Ce = \frac{q_e}{q_{max}K} + \frac{C_e}{q_{max}} \qquad \text{Equation (4)}$$

where **Ce** was the equilibrium concentration of heavy metal; **qe** was the amount of metal ions removed equilibrium; **qm** was the maximum adsorption capacity and **KL** was the Langmuir constant related to the affinity binding site. The constants **qm** and **KL** can be determined from the intercept and the slope of the linear plot of **1/qe** against 1/**Ce**

The Freundlich isotherm model is expressed in

$$\log q_e = log k_f + \frac{1}{n}\log C_e \qquad \text{Equation (5)}$$

Kf and n represent the adsorption capacity and the adsorption driving force, respectively. It has been established that as the Kf increases, the adsorption capacity increases including adsorption strength (n). a plot of log qe against log Ce was used to determine the values of n and Kf.

The values of Kf were interpreted using; for good adsorption, the estimated values of n ranged between 2–10 with 1–2 for moderate and <1 for poor adsorption (Hamzat et al., 2019)

3.11 Quality Assurance

The following quality assurance measures were considered.

3.11.1 Quality assurance on synthesis of ACNTs

1. High purity graphite powder was used for synthesis

2. Accurate temperatures were maintained throughout the synthesis process
3. Reaction time was maintained to ensure desired properties and structure of synthesized carbon nanotubes

3.11.2 Quality assurance on characterization of ACNTs

The following quality assurance was maintained to ensure optimum SEM imaging: Standard settings for Zeiss Sigma such as 55kV acceleration voltage, working distance range of 9.12 - 9.18 mm and magnification range of 284 X – 3.58 KX was done. 4 µm – 100 µm pattern that is compactible to magnification range were also chosen. Beam was assessed to ensure alignment and saturation.

Bruker FTIR with OPUS was calibrated with water vapour and spectral features was verified with expected spectral feature of water. Calibration procedure was repeated for three times to ensure accuracy of spectral features of ACNTs.

Wavelenght SPCORD 200 PLUS model 223E1451 was calibrated using didymium glass for verification. Photometric accuracy was certified with potassium dichromate and stray line level was verified with potassium chloride to ensure accurate and reliable results
X'Pert3 Powder PANalytical X-ray diffractometer was calibrated wit NIST SRM 640 (Si powder) while the wavelength, detector sensitivity and sample positioner were adjusted to ensure accuracy.

3.11.3 Quality assurance on heavy metal concentration determination

Calibration standard from Merk was prepared according to ISO/IEC 17025 guidelines by diluting single-element standard. Five different standard concentrations were prepared, and calibration curve was plotted. Calibration curve was verified by analyzing a quality control sample with known concentration to ensure accuracy. The detection limits were recorded between 0.0015 - 70 mg/L. Analyses were repeated and mean concentrations were documented

CHAPTER FOUR

RESULTS AND DISCUSSION

This subdivision presents the results of the test performed to evaluate the success of ACNTs in removing these toxic metals from water. The data obtained from various characterization techniques, including SEM, XRD, FTIR and UV-Vis were analyzed to understand the sound structure, and surface properties of the manufactured ACNTs. Results of adsorption experiments were presented in terms of adsorption capacity, removal percentage, and isotherm models.

The capacity of ACNTs as an effective adsorption material for the elimination of heavy metals from groundwater. The results are discussed in the context of the adsorption mechanisms, and kinetics. The discoveries of this schoolwork have substantial suggestions for the progress of cost-effective and competent technologies for the remediating metal-contaminated groundwater.

4.1 Images of synthesized Activated Carbon Nanotubes from SEM

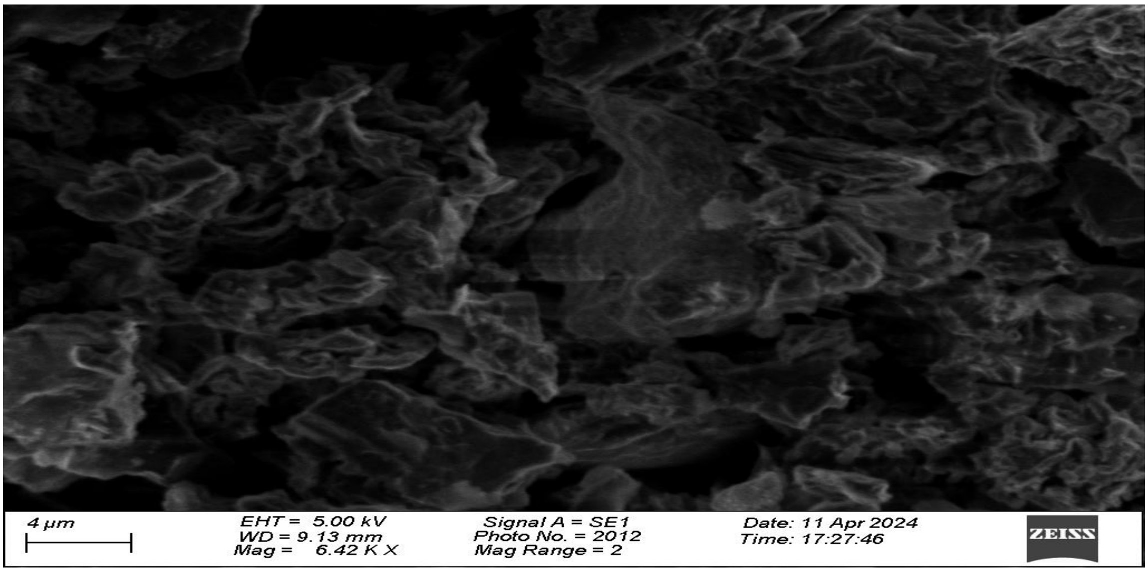

Figure 4.1 a) Image of Activated Carbon Nanotubes. Mag.= 6.42KX and 4 µm

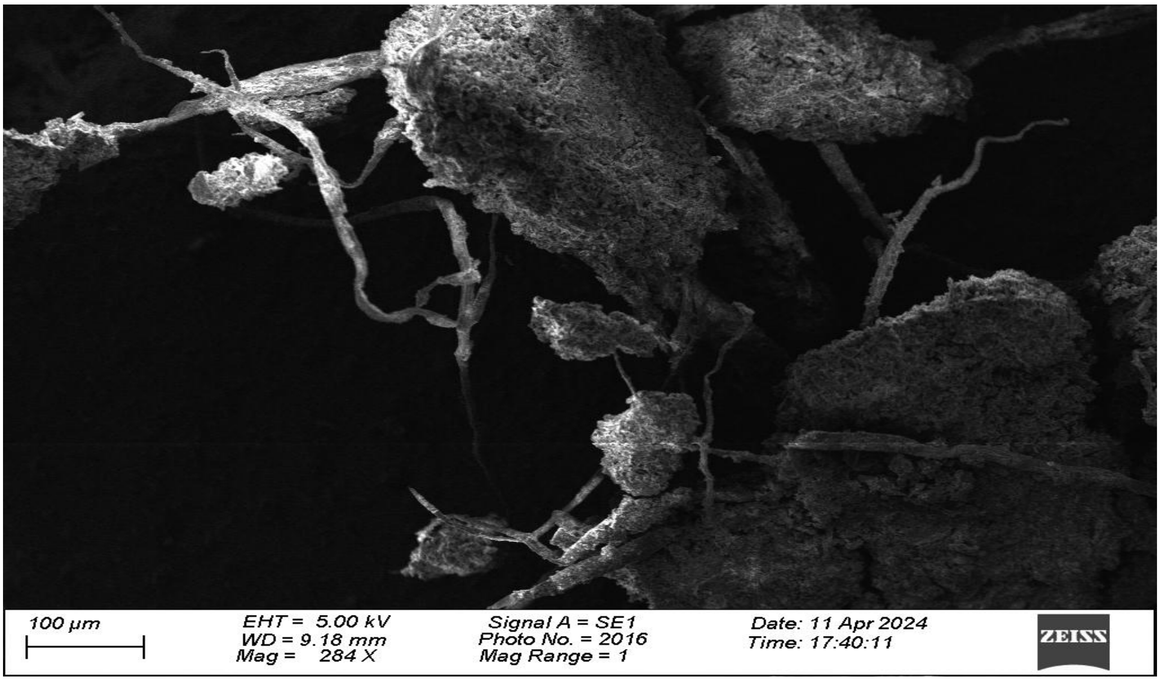

Figure 4.1 b) Image of Activated Carbon Nanotubes a Mag.= 248X and 100 µm

Figures 4.1 a) and b) depict images of Activated carbon nanotubes with magnification range of 284 X to 6420 X. The higher magnification range help detailed study of particle size and surface features of activated carbon nanotubes which influence their properties. The particle sizes were found between 0.0553 µm (55.3 nm) to 4.480 µm (448 nm) with mean particle size of 254. This range indicates polydisperse distribution of carbon nanotubes, which agrees with (Liu et al., 3013; Singh et al., 2018). Image show web-like carbon nanotubes with holes between them (Itas et al., 2020). Surface structure signifies twisted and indiscriminately oriented web-like network, more than a few micrometers long, screening that the reduced powders keep the shape of the reduction vessel (Aslam et al., 2021). It was also found that, ACNTs have solidity range between 0.83 to 0.92 which indicates that 83% to 92% of the activated carbon nanotubes surface are occupied by carbon atoms with 8% to 17% surface defects and impurities. This indicates moderate level of defects (Wang et al. 2017).

4.2 FTIR Results of Activated carbon nanotubes

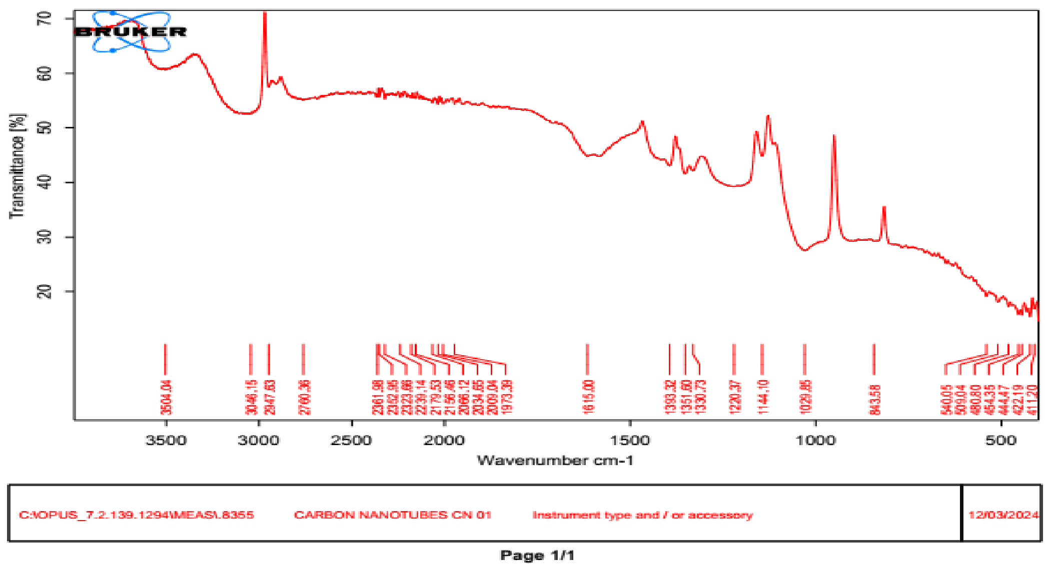

Fig. 4.2 IR spectrum of Activated carbon nanotubes

FTIR analysis was performed to characterize the surface functional groups, defects, and impurities. Spectral lines range from 4000 cm^{-1} to 400 cm^{-1}. The peaks recorded at specific wave numbers give valuable information on specific functional group connected to the superficial of carbon.

From figure 4.2, major peaks were recorded 3504.04 cm^{-1}, 3046.15 cm^{-1}, 2947.63 cm^{-1}, 1615 cm^{-1} - 1393.32 cm^{-1}, 1220.37 cm^{-1} – 1144.10 cm^{-1}, 1029.85 cm^{-1} and 843.58 cm^{-1}. The broad peak at 3504.04 cm^{-1} suggests O-H stretching vibration which suggest presence of hydroxyl (OH) group which may be due to absorbed water molecules on the surface of activated carbon nanotubes (Coates, 2000). Sharp peak at 3046.15 cm^{-1} and 2947.63 cm^{-1} correspond to C-H stretching vibration suggest the presence of alkene. The peaks 1615 cm^{-1} - 1393.32 cm^{-1} suggest C=C stretching vibration in activated carbon nanotubes. This is a characteristic band in line with aromatic rings of rolled graphene layers (Brijesh Gaud, Amrita Singh, 2019) (Aslam et al., 2021). Two pointing peaks between 1220.37 cm^{-1}-1144 cm^{-1} correspond to C-O stretching vibration. Peak at 843 cm^{-1} is assigned a C-H bending vibration of an alkyl group. The presence of C=C, C-H and C-O affects the reactivity, dispersion, and the interactions of carbon nanotubes with other materials.

4.3 UV vis results of activated carbon nanotubes

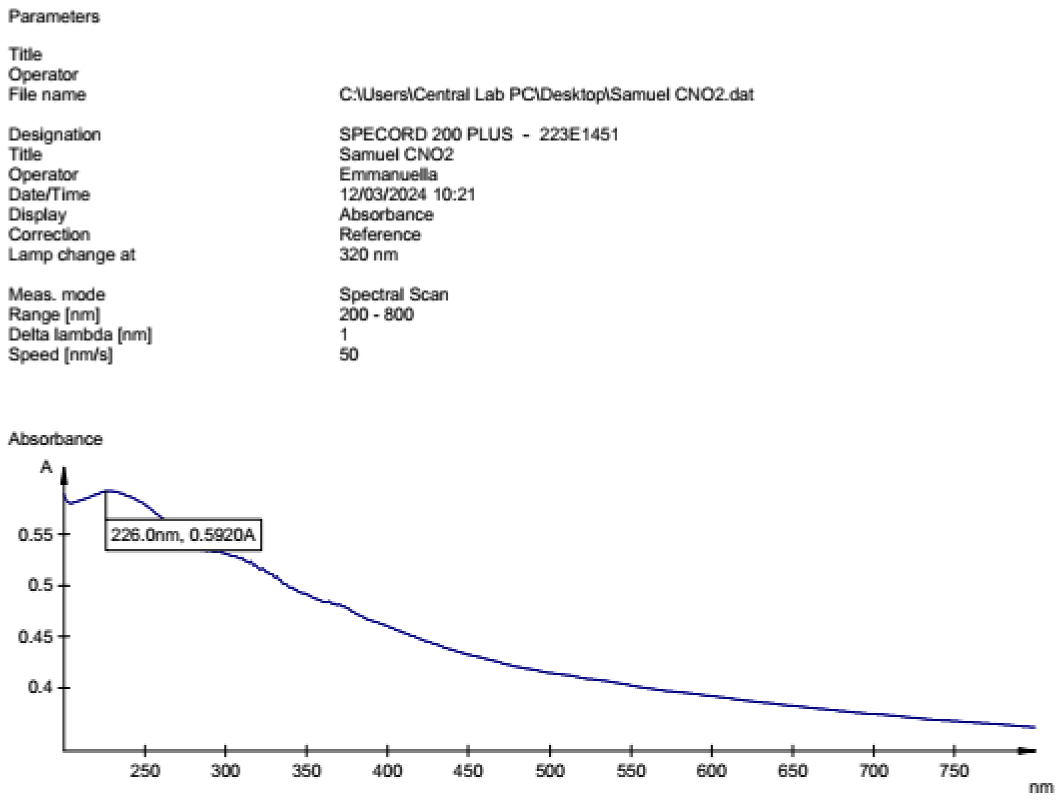

Figure 4.3: UV spectrum of activated carbon nanotubes

Optical features of activated carbon nanotubes was determined using the SPECTROCORD 200 PLUS with spectrum range of 200-800 nm. The spectrum revealed broad absorption peak at 226.0 nm with an absorbance of 0.592 A. the absorption peak can be attributed to the electronic transitions in carbon nanotubes which are influenced by their diameter, structure, and functionalization (Kataura et al., 1991). The broad peak at 226.0 nm may indicate the presence of π- π transitions in the activated carbon nanotubes which are characterized by sp2 hybridized carbon atoms in the ACNTs structure (Chen & Collier, 2005). The π-plasmon resonance of ACNTs, indicating that there is likely to be existence of graphitic carbon (Kataura et al., 2009).

Absorbance value (0.5920) suggests a moderate concentration of ACNTs (Liu et al., 2011).

The activated carbon nanotubes is likely to be multiwalled due to the broader absorption peaks, including the 226.0 nm peak (Li et al., 2013).

4.4 XRD Results of Activated carbon nanotubes

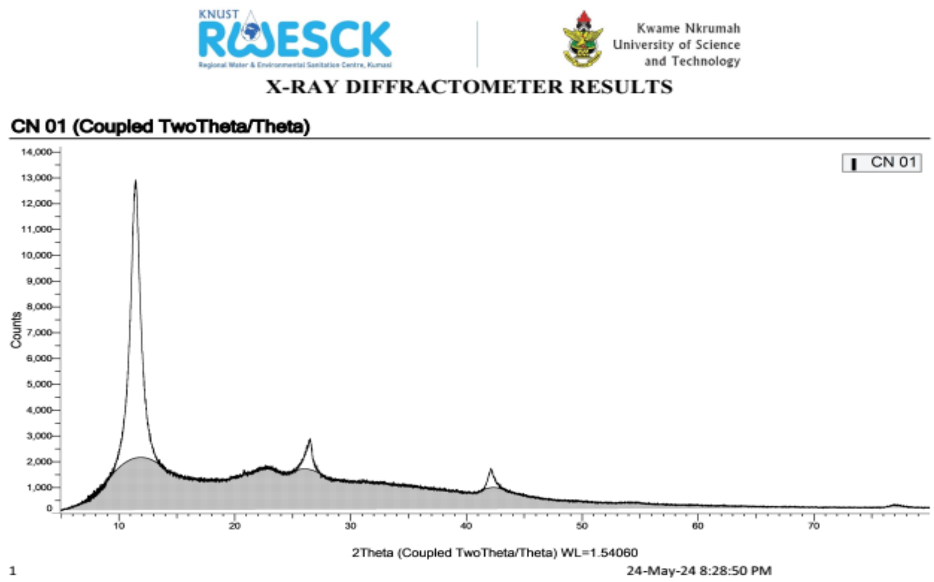

Figure 4.4: XRD spectrum of activated carbon nanotubes

Pattern in figure 4.4 show X-ray diffractometer results for Activated carbon nanotubes. This provides valuable information about their structural and qualitative properties such as crystallinity, types, fingerprints, and quantitative material (Abdulrazzak et al., 2019) The XRD spectrum depicts three diverse peaks at 11.985°, 26.201°, and 42.134° (Hussain, 2014). These peaks correspond to specific crystal planes in the carbon nanotube's structure. The peak at 11.985° is likely attributed to the plane, which is a characteristic peak for CNTs. This peak indicates the presence of multi-walled carbon nanotubes (Liu et al. 2018) and **(**Ren et al 2015).

Peak at 26.201° corresponds to the (100) plane, which is also a common peak for CNTs. This peak suggests the presence of single-walled carbon nanotubes (SWCNTs) or double-walled carbon nanotubes (MWCNTs) (Kim et al. 2019). The distinct peaks suggest that carbon nanotubes comprise of both single walled and multiwalled carbon nanotubes. The sharp peak at 26.46° conforming to specific repeating three-dimensional arrangement of carbon in activated carbon nanotubes. (Brijesh Gaud and Amrita Singh, 2019). This was depicted in the SEM where web-like structure of carbon nanotubes was shown.

The peak at 42.134° is attributed to the plane, which is another characteristic peak for CNTs. This peak further supports the occurrence of MWCNTs, (Zhang et al. 2020) X-ray pattern indicates moderate level of crystallinity, suggesting that the ACNTs have a relatively ordered structure. However, the presence of broad peaks and a high background noise level may indicate some degree of disorder or impurities in the sample (Li et al. 2017)

In conclusion, the XRD results suggest that the activated carbon nanotubes present in the sample are likely a mixture of MWCNTs, FWCNTs, and SWCNTs. The presence of characteristic peaks at 11.985°, 26.201°, and 42.134° supports this conclusion.

4.5 Adsorption Studies on activated carbon nanotubes

Adsorption studies was performed to establish conditions for highest adsorption of metal pollutants. Impact of contact time and quantity of adsorbate was examined.

4.5.1 The effect of contact time on removal of Pb, As and Cd.

Tables 4.1 (a), (b) and (c) depict contact and percentage of metal concentration removed by 0.1 g of activated carbon nanotubes at 6.8 pH and stirring speed of 6.5 rpm at 25°C temperature. In each experiment, 0.01 $molL^{-1}$ standard solution of metal ion was used.

Table 4.1 (a). The effect of Contact time on percentage of Pb removed.

Contact time (Minutes)	10	20	30	40	50	60
% of Pb removed	55	78	89	88	89	89

Table 4.1 (b): The effect of contact time on Percentage (%) of As removed

Contact time (Minutes)	10	20	30	40	50	60
% of As removed	62	71	90	93	93	93

Table 4.1 (c): The effect of contact time on Percentage (%) of Cd removed

Contact time (Minutes)	10	20	30	40	50	60
% of Cd removed	66	74	91	91	91	91

From tables 4.1 (a), (b) and (c), rise in contact time increase rate of elimination of the three metals due to increase in the interaction between carbon nanotubes and metals.

More of the heavy metals get opportunity to diffuse onto carbon nanotubes (Gupta et al, 2019). Optimum elimination potential for each metal was recorded between 30 – 40 minutes, where equilibrium is reached (Khoshmardan et al., 2021).

4.5.2. The effect of amount of Carbon nanotubes on removal of Pb, As and Cd

The amount of carbon nanotubes required to remove maximum quantity of heavy metals was investigated to ascertain the maximum amount required optimum adsorption. In each experiment, 0.01 $molL^{-1}$ standard solution of metal ion was used at a pH of 6.8, 6.5 rpm of at 25 ^{o}C temperature and contact time of 30 minutes. The results were recorded and presented below.

Table 4.2.(a) Table showing the outcome of quantity of adsorbate on Percentage (%) of Pb removed

Quantity of carbon nanotubes (g)	0.1	0.2	0.3	0.4	0.5
% of Pb removed	71	78	90	90	90

Table 4.2.(b) Table showing the outcome of quantity of adsorbate on Percentage (%) of As removed

Quantity of carbon nanotubes (g)	0.1	0.2	0.3	0.4	0.5
% of As removed	72	84	89	90	90

Table 4.2.(c) Table showing the consequence of quantity of adsorbate on Percentage (%) of Cd removed

Quantity of carbon nanotubes (g)	0.1	0.2	0.3	0.4	0.5
% of Cd removed	69	74	82	82	82

From tables 4.2(a), (b) and (c), increasing the amount of Activated carbon nanotubes significantly increase metal adsorption. Increasing quantity of carbon nanotubes provide larger surface area for metal ion to attach. Equilibrium was reached around 0.2 g- 0.3 g. (Wang et al, 2019).

4.6 Concentrations of Pb, As and Cd in water from hand-dug wells

Table 4.3: Mean concentrations of Pb, As and Cd in Nsapor Well before and after treatment in mg/L

Source	Metal	Mean conc. b/4 treatm.	SD	Mean conc. A/F treatm	SD	Metal Removed	SD	% Removed
Nsapor	Pb	0.0062	0.00018	0.00016	0.00012	0.006	0.0011	97.42
	As	0.0065	0.00113	0.00015	0.00011	0.0063	0.0013	96.92
	Cd	0.0046	0.0010	0.00016	0.00012	0.0044	0.0019	96.52

Table 4.3. presents the average levels of Pb, As and Cd in water from Nsapor Well water before and after treatment with carbon nanotubes. For Pb, 0.0062 mg/L initially recorded was with 0.00018 standard deviation and the level after treatment was 0.00016 mg/L with standard deviation of 0.00012. This shows an estimated amount 0.006 mg/L of Pb removed which represents a significant percentage of 97.42% (Rashid, 2016). Although

the initial level of Pb in the sample was below the WHO limit for drinking water but there is great removal of Pb in water sample by carbon nanotubes.

The study also revealed that, the initial level of As in water was 0.0065 mg/L and was reduced to 0.00015 mg/L. this means that 0.0063 mg/L of As was removed from water sample representing 96.92% of the initial level. The initial level of the metal was below the WHO limit but treatment with carbon nanotubes further reduced it. (Li et al., 2022) The initial level of Cd from the study gave 0.0046 mg/L (SD 0.001) and was determined to be 0.00016 mg/L (SD 0.00012) after treatment with carbon nanotubes. The initial level of Cd exceeds the WHO limit for drinking water (WHO, 2017). Carbon nanotubes reduced the level below the WHO limit with a percentage reduction of 96.52%. Summary of findings in table 4.3.(a) is represented in figure 4.5.1 below.

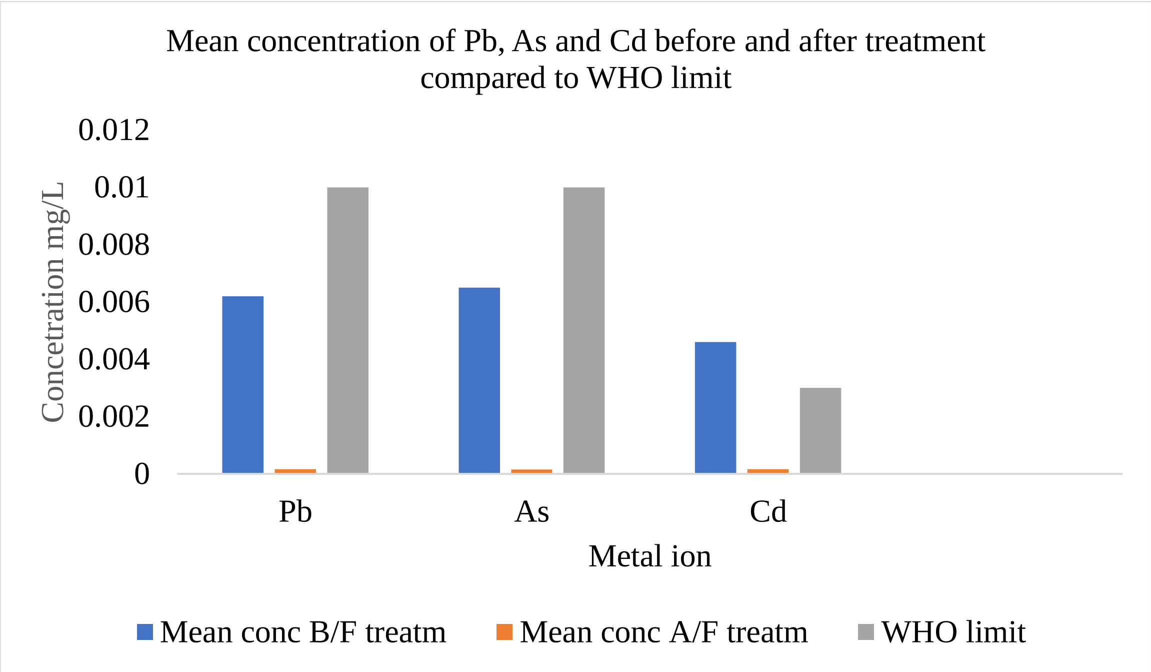

Figure 4.5: Mean level of Pb, As and Cd in Nsapor well before and after treatment compared to WHO limit

Table 4.4: Mean concentrations of Pb, As and Cd in Biadan Well water before after treatment in mg/L

Source	Metal	Mean conc. b/4 treatm.	SD	Mean conc. A/F treatm	SD	Metal Removed	SD	% Removed
Biadan	Pb	0.0025	0.0012	0.00015	0.0001	0.0023	0.0012	94.00
	As	0.0046	0.00017	0.00018	0.0001	0.0044	0.0023	95.65
	Cd	0.0028	0.0015	0.00015	0.0001	0.0020	0.0013	92.86

The results presented in table 4.4 demonstrate efficiency of activated carbon nanotubes in treating Pb, As and Cd in Biadan well water. The mean level of metal ions significantly reduced after treatment. The mean concentrations recorded before treatment for Pb, As and Cd were 0.0025 mg/L, 0.0046 mg/L and 0.0028 mg/L respectively with their respective standard deviations of 0.0012, 0.00017 and 0.0028.

These levels of metals ions are all less than WHO limits for water. (WHO, 2017). Mean levels of Pb, As and Cd recorded after treatment with carbon nanotubes were recorded as 0.00015 mg/L, 0.00018 mg/L and 0.00015 mg/L correspondingly. These values are significantly lower that WHO limit for each metal ion permitted for drinking water. Activated carbon nanotubes efficiently removed 94% of Pb, 95.64% of As and 92.86% of Cd (Khoshmardan et al., 2021). These values show significant efficiency of carbon nanotubes in removing these heavy metals from drinking water (Baby & Saifullah, 2019). Figure 4.6 presents summary of the impact of carbon nanotubes on these selected heavy metals in Biadan well water.

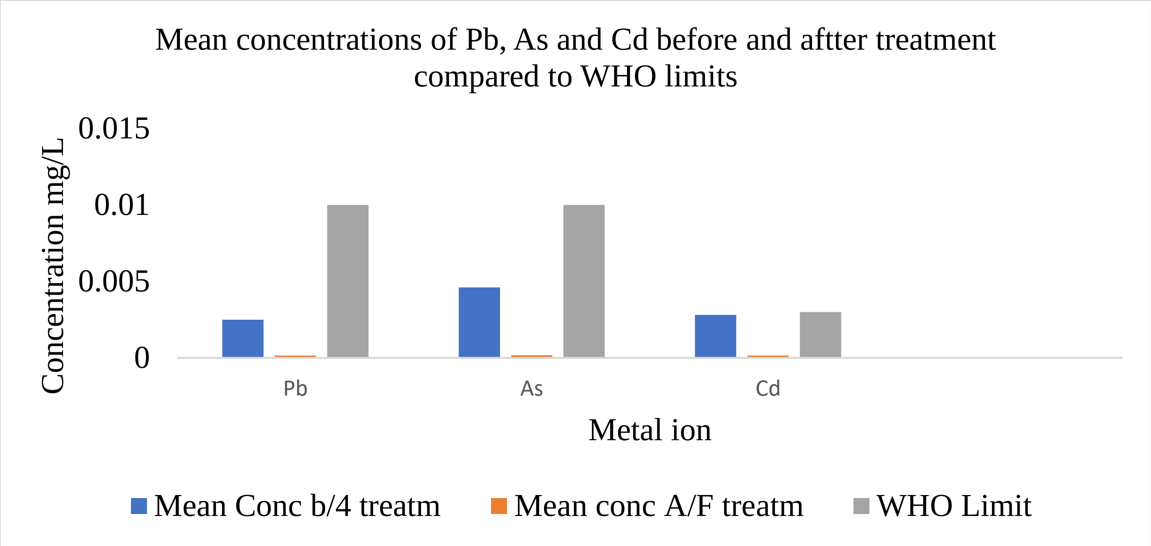

Figure 4.6: Mean levels of Pb, As and Cd in Biadan Well before and after treatment compared to WHO limits.

Table 4.5: Mean concentrations of Pb, As and Cd in Koraso Well water before and after treatment in mg/L

Source	Metal	Mean conc. b/4 treatm.	SD	Mean conc. A/F treatm	SD	Metal Removed	SD	% Removed
Koraso	Pb	0.0030	0.0012	0.00016	0.0001	0.00280	0.0013	94.67
	As	0.0038	0.0011	0.00014	0.0001	0.0037	0.006	96.32
	Cd	0.0025	0.0014	0.00016	0.0001	0.0023	0.0013	93.60

Table 4.5 presents the mean levels of metals in Koraso well before and after intervention. The mean concentrations recorded were Pb, 0.0030 mg/L with standard deviation of 0.0012, As, 0.0030 mg/L with standard deviation of 0.0011 and Cd 0.0025 mg/L with standard deviation of 0.0014. Levels recorded suggest water is moderately polluted with the level below WHO limits Pb 0.01 mg/L, As, 0.01 and Cd 0.003 mg/L.

After intervention, the concentration of Pb, As and Cd were decreases to 0.00016 mg/L, 0.0014 mg/L and 0.00016 mg/L with standard deviations of 0.0001 for each metal ion respectively as presented in Figure 4.7

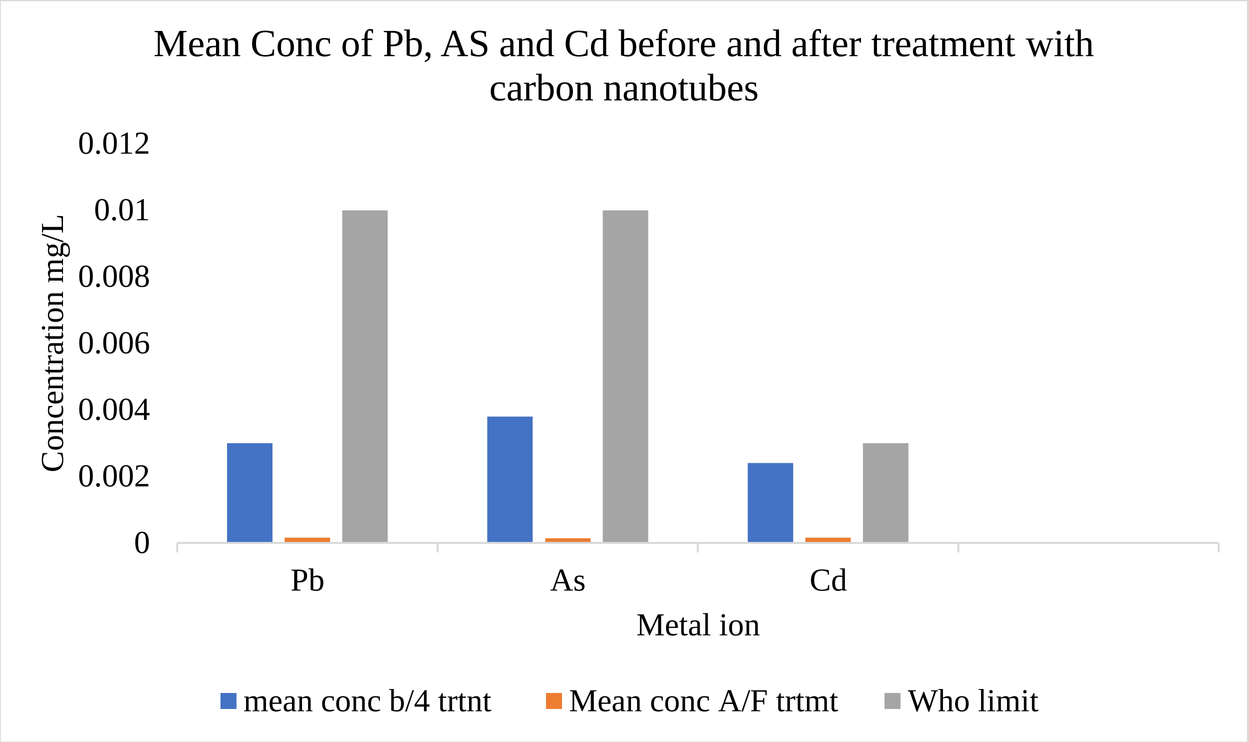

Figure. 4.7: Mean levels of Pb, As and Cd in Koraso Well before and after treatment with Activated carbon nanotube compared to WHO limits

Although water is moderately polluted with the metals, the carbon nanotubes show very high removal rate for Pb, As and Cd. Efficiencies were 94.67%, 96.32% and 93.60% Lead, Arsenic and Cadmium respectively (Ma et al., 2017).

The application of carbon nanotubes has resulted in significant reduction of the concentrations of metal ions in water samples with removal efficiency above 93% for all the metals. The studies suggest that Activated carbon nanotubes are efficient remedy for Pb, As and Cd (Li et al 2018)

4.7 Isotherms

Adsorption technology has appeared as a promising resolution for eradicating pollutants from water with Activated carbon nanotubes as an effective adsorbent material due to its unique properties. Adsorption isotherms play impact significantly in explaining the interaction between Carbon nanotubes and the heavy metal ions. Isotherms define the connection between the amount of adsorbate (metal ions) and Carbon nanotubes (adsorbent) at equilibrium.

4.7.1 Langmuir Isotherm

Highest removal efficacy of Activated carbon nanotubes and affinity between each metal and carbon nanotube using Langmuir equation; $\frac{1}{q_e} = \frac{1}{Ceq_{max}K} + \frac{C_e}{q_{max}}$. The fit Langmuir model in explaining insight of the adsorption process was also estimated with coefficient of determination (Dehmolaei & Vadi, 2014)

Table 4.6: Langmuir Adsorption Isotherm on Pb

Sample	q_e	Ce	$1/q_e$	1/Ce	q_{max}	K	R^2	X^2
Nsapor	0.0062	0.00016	166.667	6250.00	5.375mg/L	0.02678	0.523	0.00
Biadan	0.0025	0.00015	434.783	6666.667				0.00
Koraso	0.0030	0.00016	357.143	6250.00				0.00

In inspection of values from table 4.6.1, show that carbon nanotubes can absorb maximum of 5.375 mg/L of Pb in water samples. This shows significant amount which can bring metal ion concentration of more polluted water below WHO limit. Langmuir constant, K value of 0.02678 moderate energy of adsorption of Pb. R^2 value recorded as 0.523 suggest that Langmuir model moderately fits the adsorption model. This indicates that there was approximately 53.2% of variance adsorption data can be explained by

Langmuir model. Chi square statistics is showing excellent fit of Langmuir model with an X^2 value of 0.00. According to Foo and Hameed, (2010), R^2 values may indicate a complex adsorption process and suggest that the process may be influenced by multiple factors, Langmuir model cannot fully account for the adsorption process.

Table 4.7: Langmuir Adsorption Isotherm on As

Sample	q_e	Ce	1/qe	1/Ce	q_{max}	K	R^2	X^2
Nsapor	0.0063	0.00015	158.730	6666.667	1.327mg/L	0.0753	0.027	0.00627
Biadan	0.0044	0.00018	227.273	5555.556				0.00436
Koraso	0.0037	0.00014	270.270	7142.857				0.00366

Experimental data was analyzed and presented in table 4.7 to investigate the affinity of As to Carbon nanotubes as well as its adsorption energy suggest that As has moderate affinity to carbon nanotubes as suggested by its K value of 0.0753. There was also a reduced maximum amount of adsorption 5.375 mg/L in Pb to 1.372 mg/L in As. This has shown significant decrease in the maximum quantity of As per unit mass of carbon nanotubes that can be adsorbed. Langmuir model moderately fits the adsorption of As as depicted R^2 value 0.027 (Sweetman, 2012)

Table 4.8: Langmuir Adsorption Isotherm on Cd

Sample	q_e	Ce	1/qe	1/Ce	q_{max}	K	R^2	X^2
Nsapor	0.0044	0.00016	227.273	6250.00	2.5664	0.3896	0.469	0.004
Biadan	0.0020	0.00015	500	6666.667				0.0017
Koraso	0.0023	0.00016	434.783	6250.00				0.0017

Highest removal capacity was found to be 2.5664 mg/L indicating the amount of Cd that can be adsorbed per unit mass of carbon nanotubes at saturation compared to Foo et al 2020. K values estimated was 0.3896 as shown in table 4.8 suggest moderate affinity of Cd onto carbon nanotubes as reported by (Sweetman, 2012). R^2 for Langmuir model was 0.469, indicating moderate agreement of the model to investigational values. This model can provide some insight into the adsorption of Cd but may not capture detailed underlying mechanisms of the system.

4.7.2 Freundlich Isotherm

Suitability of the model in describing the elimination process of target impurities was tested. The estimated parameters provided appreciated understandings into the adsorption mechanism and affinity of carbon nanotubes for the selected metals in the sample using $\log q_e = \log k_f + \frac{1}{n}\log C_e$ (Dehmolaei & Vadi, 2014).

Table 4.9: Freundlich Isotherm on Pb.

Sample	q_e	Ce	Log q_e	Log Ce	Kf	n	R^2	X^2
Nsapor	0.0062	0.00016	-2.208	-3.796	29.7	0.118	0.492	10.545
Biadan	0.0025	0.00015	-2.602	-3.824				10.633
Koraso	0.0030	0.00016	-2.523	-3.796				10.551

From table 4.9, the results indicate varying adsorption capacities and intensities. Freundlich constant, 29.7 indicates higher adsorption of Pb in samples onto carbon nanotubes. N value of 0.118 suggest an unfavorable adsorption process which may be attributed to the heterogenous nature of activated carbon nanotubes as reported by Erdogan, 2018 . Chi square error value of between 10.545-10.633 were recorded from each sample site which depicts the a better fit of Pb data onto different samples (Dehmolaei & Vadi, 2014).

Table 4.10: Freundlich Isotherm on As

Sample	q_e	Ce	Log q_e	Log Ce	Kf	n	R^2	X^2
Nsapor	0.0063	0.00015	-1.201	-3.823	0.8	1.471	0.961	0.00285
Biadan	0.0044	0.00018	-2.357	-3.745				0.0008580
Koraso	0.0037	0.00014	3.432	3.854				0.000856

Table 5.10. depicts the results from application of Freundlich model on adsorption data of As. Freundlich constant was 0.8 which suggest that Freundlich isotherm has higher efficiency in removal of As (Dehmolaei & Vadi, 2014). 1/n was 0.6798 depicts that favorable adsorption of As and the process occurred on heterogenous activated carbon nanotubes (Erdogan, 2018). R^2 of 0.961 propose this model can fully explain the process involved in As remediation (Saleh et al., 2024)

Table 4.11: Freundlich Isotherm on Cd

Sample	q_e	Ce	Log q_e	Log Ce	Kf	n	R^2	X^2
Nsapor	0.0044	0.00016	-3.3357	-3.796	36.38	0.125	0.106	12.21
Biadan	0.0020	0.00015	-2.699	-3.823				12.08
Koraso	0.0023	0.00016	-2.538	-3.796				12.21

Table 4.11 presents the following from Freundlich model; Freundlich constant, Kf of 36.38 which suggest that carbon nanotubes have higher absorption capacity of cadmium. On the other hand, n was recorded as 0.125 which suggest that adsorption process of Cd

is fairly favorable due to heterogenous multiparticle nature of carbon nanotubes (Mallakpour & Khadem, 2019).

In Summary, synthesized Activated carbon nanotubes has exhibited effective redress for the metal remediation. Elimination ability was best explained by different isotherm model resulting from different interaction between metal ion and Activated carbon nanotubes. Elimination of Pb and Cd were best explained by Langmuir Isotherm while adsorption of As best fitted Freundlich isotherm model (Osikoya et al., 2014)

CHAPTER FIVE

SUMMMARY, RECOMMENDATIONS AND CONCLUSION

5.1 Introduction

This section summarizes the study's key findings and implications. The research investigated carbon nanotubes' potential as effective adsorbents for removing Pb, As, and Cd from groundwater. The study's objectives were to:

1. Synthesize activated carbon nanotubes from graphite powder
2. Evaluate carbon nanotubes' adsorption capacity.
3. Determine optimal adsorption conditions.
4. Apply adsorption models to explain capacity.

The findings aim to contribute to a harmless, convenient, cost-effective, and environmentally friendly method for treating drinking water, leveraging modern knowledge to remove heavy metals and other contaminants that conventional methods struggle to eliminate.

5.2 Key Findings

The study's findings revealed that activated carbon nanotubes (ACNTs) were successfully synthesized from graphite powder, with particle sizes ranging from 55.3 nm to 480 nm. Characterization confirmed the presence of multi-particle ACNTs, including single-walled and double-walled carbon nanotubes, which enhanced their adsorption capacities. The ACNTs demonstrated significant potential for removing Pb, As, and Cd from groundwater, achieving removal rates of 93.6% to 97.42%. Adsorption experiments and isotherm models provided insights into the removal efficiencies and mechanisms. These findings have important implications for developing efficient and sustainable technologies for heavy metal removal, suggesting that ACNTs are a promising approach for improving water quality and mitigating risks associated with heavy metal contamination.

5 3 Conclusion

This study aimed to provide clean and safe water by synthesizing activated carbon nanotubes (ACNTs) from graphite powder. Instrumental analysis revealed ACNTs with particle sizes (55.3 nm - 448 nm) and surface areas (140.68 $\mu m^2/g$ - 73,923 $\mu m^2/g$), exhibiting moderate crystallinity and unique properties that enhance adsorption capacity. The study investigated the impact of pH, adsorbate quantity, and contact time on ACNTs' adsorption capacity. Optimum conditions were:

- pH 6.5-7.5
- Adsorbate quantity: 0.3-0.5 g
- Contact time: 20-40 minutes

ACNTs demonstrated high adsorption capacities:

- Pb: 97.4%
- As: 96.92%
- Cd: 96.53%

Langmuir model best described Pb and Cd adsorption (monolayer adsorption), while Freundlich model suited As adsorption (chemisorption). This study confirms ACNTs' effectiveness in removing heavy metals from groundwater, highlighting their potential for water purification applications.

5.4 Recommendations

1. Comparative analysis: Evaluate activated carbon nanotubes' effectiveness against other adsorbents in removing heavy metals, highlighting advantages.

2. Sustainability assessment: Investigate reusability of activated carbon nanotubes for economic feasibility and environmental sustainability.
3. Scalability studies: Conduct experimental-scale tests to assess activated carbon nanotubes' efficacy in removing heavy metals from large water quantities for public consumption.
4. Mechanistic insights: Explore additional adsorption models and experimental conditions to deepen understanding of adsorption processes for various metals.

5.5 Future Research Directions

1. Conduct a mechanistic study to advance understanding of adsorption processes and relationship between carbon nanotubes and heavy metals.
2. Investigate the potential toxicity and ecological impact of carbon nanotubes in ground water redress and applications.
3. Explore the combination of carbon nanotubes with other technologies to for a hybrid to enhance the effectiveness and efficiency in heavy metal removal

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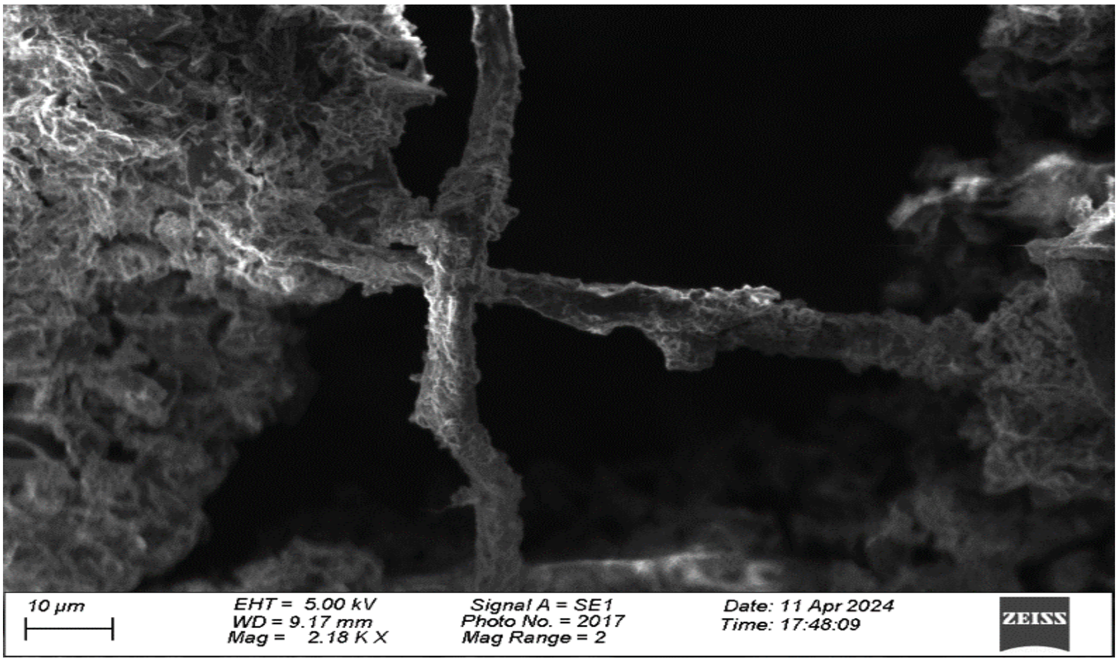
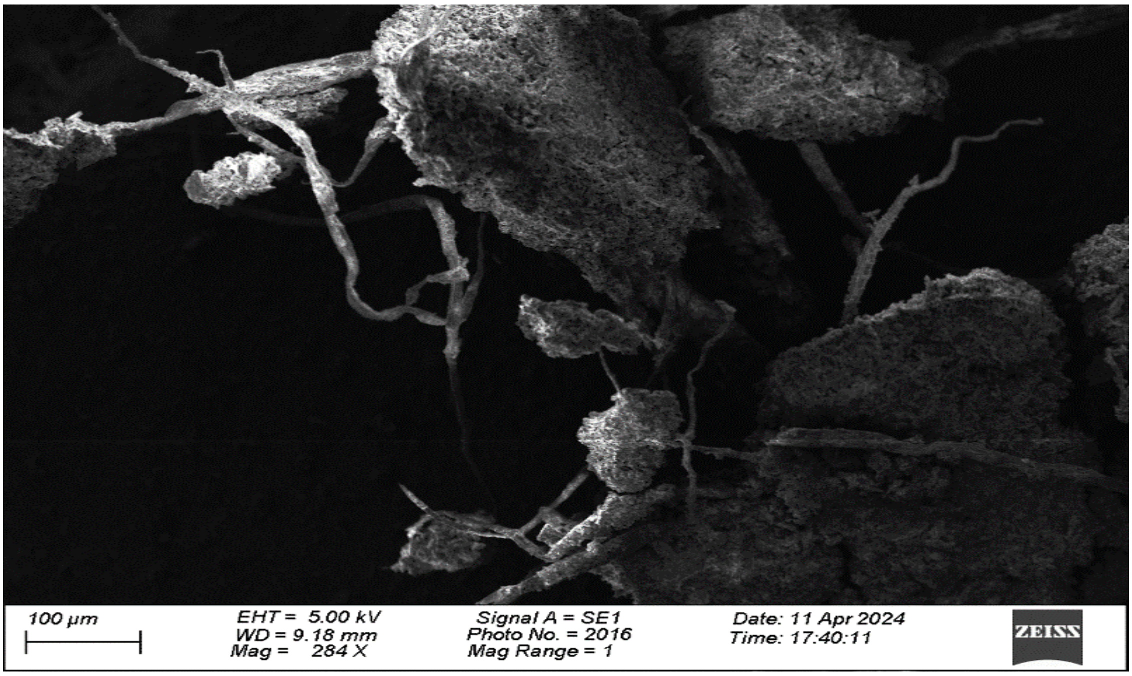
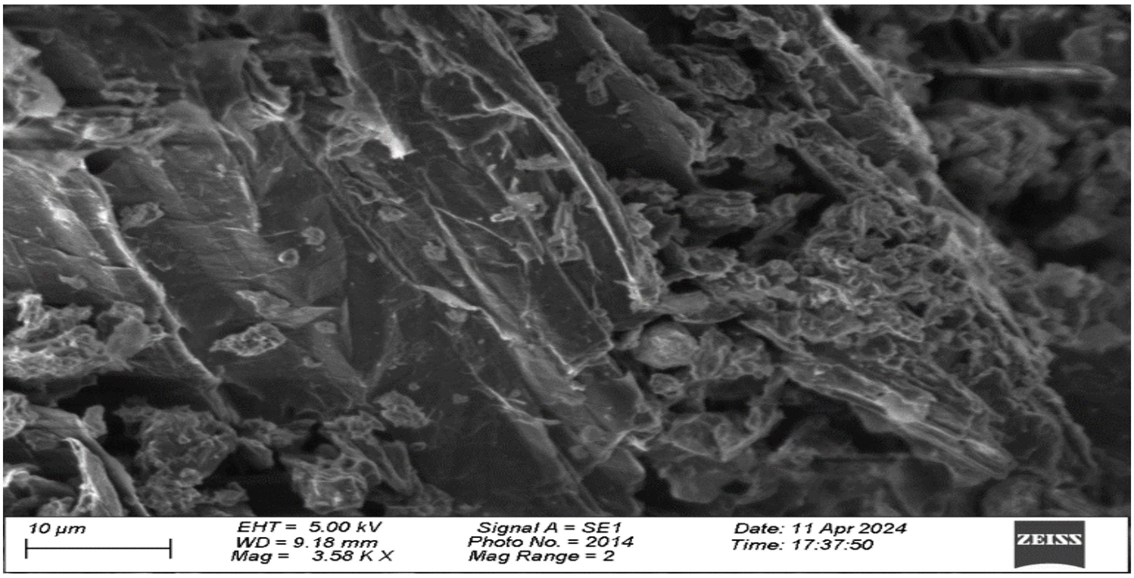
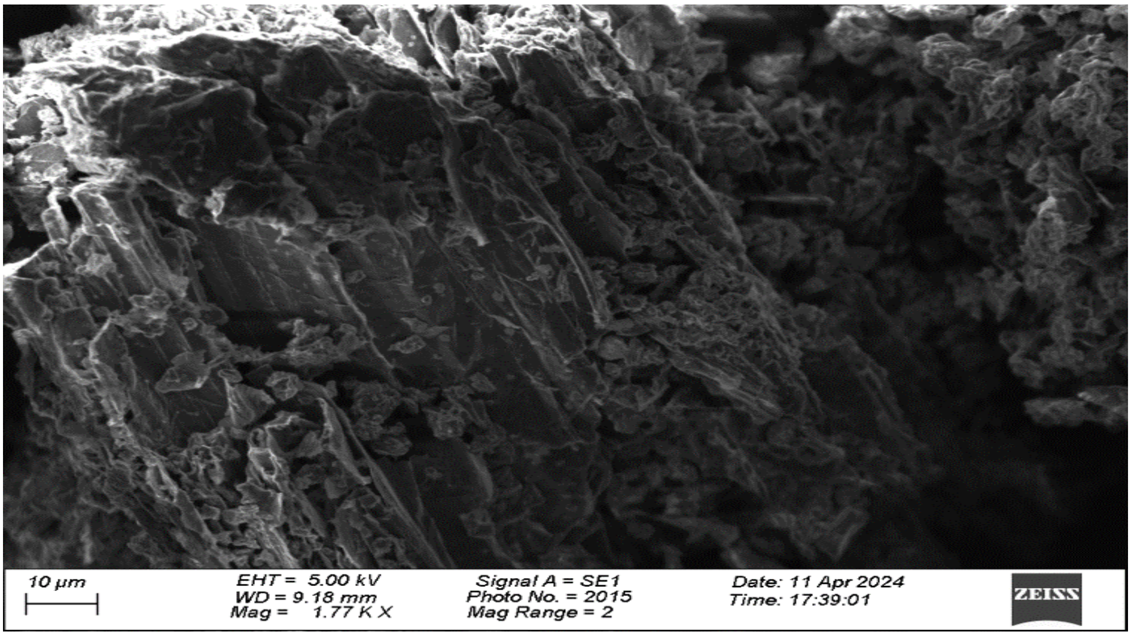
APPENDICES

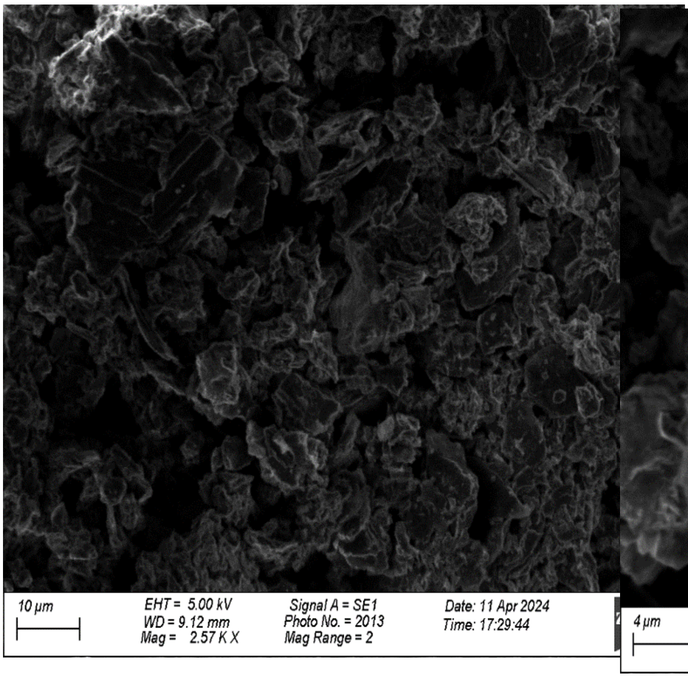
Appendix A: SEM Images of Activated Carbon Nanotubes

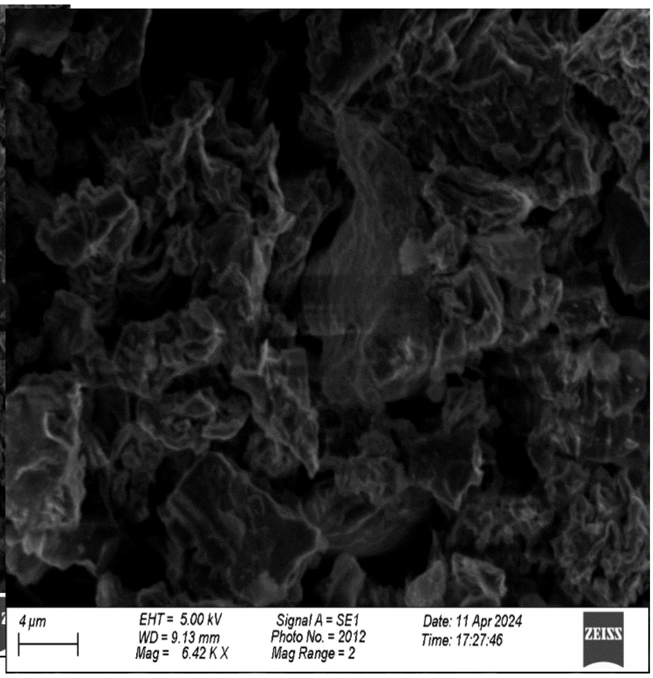





10 µm
EHT = 5.00 kV
WD = 9.18 mm
Mag = 3.58 K X
Signal A = SE1
Photo No. = 2014
Mag Range = 2
Date: 11 Apr 2024
Time: 17:37:50
ZEISS


10 µm
EHT = 5.00 kV
WD = 9.18 mm
Mag = 1.77 K X
Signal A = SE1
Photo No. = 2015
Mag Range = 2
Date: 11 Apr 2024
Time: 17:39:01
ZEISS

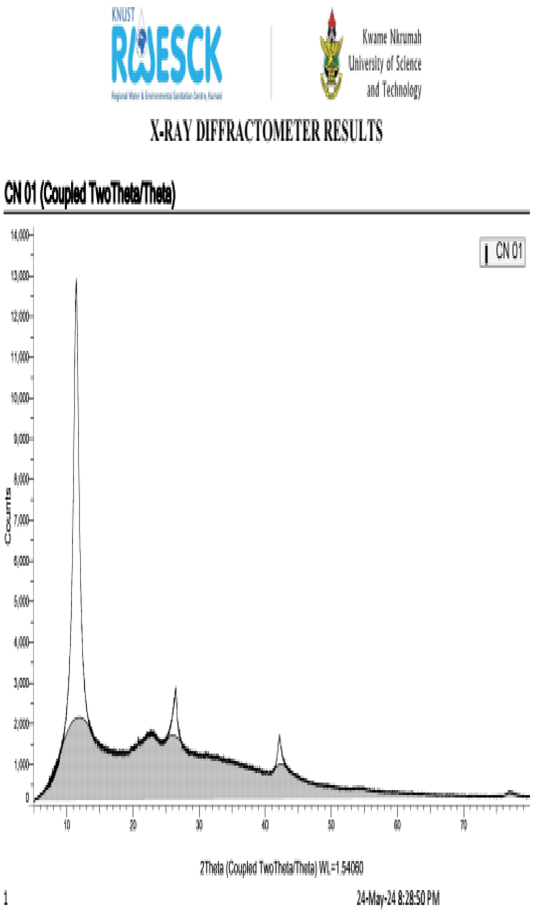

10 μm
EHT = 5.00 kV
WD = 9.12 mm
Mag = 2.57 K X
Signal A = SE1
Photo No. = 2013
Mag Range = 2
Date: 11 Apr 2024
Time: 17:29:44

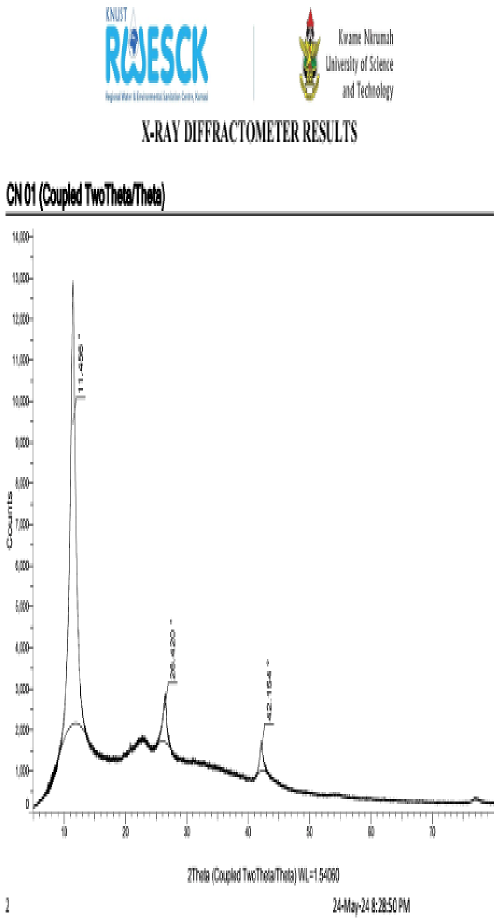

4 μm
EHT = 5.00 kV
WD = 9.13 mm
Mag = 6.42 K X
Signal A = SE1
Photo No. = 2012
Mag Range = 2
Date: 11 Apr 2024
Time: 17:27:46

Appendix B: Summary of Interpretation SEM Images

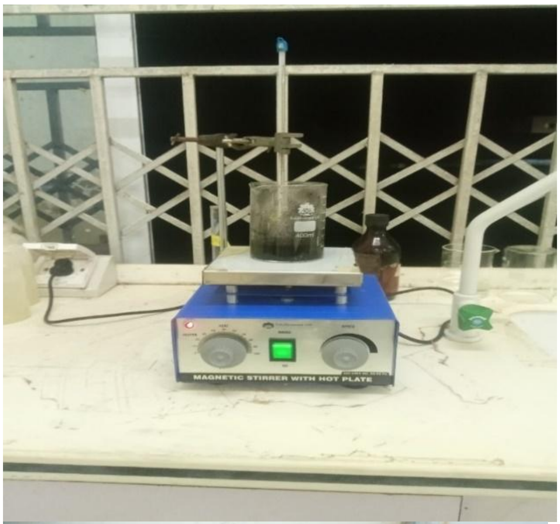
Slice	Count	Total Area	Average Size (Micrometer)	%Area	Mean
CN01_00.tif	202	140.684	0.0696	9.672	254.335
CN01_05.tif	2353	1301.631	0.0553	10.861	254.394
CN01_04.tif	1649	73923.813	4.4829	10.363	254.44
CN01_03 (1).tif	66	1843.281	2.7929	9.606	254.412
CN01_02.tif	917	1323340	14.43	16.827	254.191
CN01_01.tif	63	75535	11.988	9.605	253.618

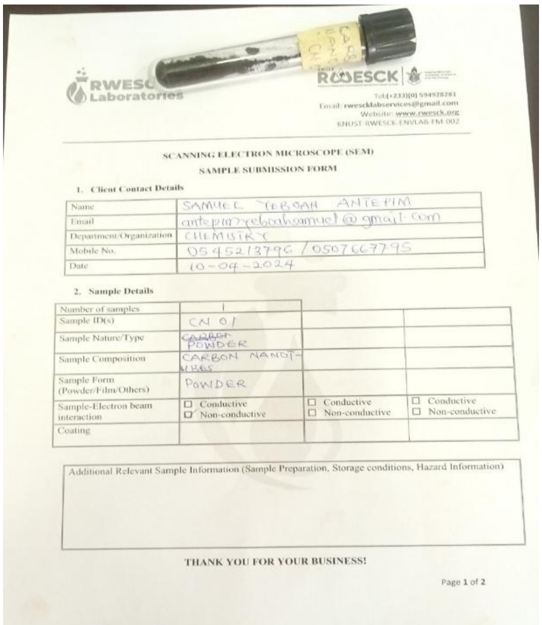
Appendix C: XRD Spectra of Activated carbon nanotubes





Appendix D: Images of Sythesis Process





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KNUST-RWESCK-ENVLAB-FM-002

SCANNING ELECTRON MICROSCOPE (SEM)

SAMPLE SUBMISSION FORM

1. Client Contact Details

Name	SAMUEL YEBOAH ANTEPIM
Email	antepimyeboahsamuel@gmail.com
Department/Organization	CHEMISTRY
Mobile No.	0545213796 / 0507667795
Date	10-04-2024

2. Sample Details

Number of samples	1		
Sample ID(s)	CN 01		
Sample Nature/Type	~~CARBON~~ POWDER		
Sample Composition	CARBON NANOTUBES		
Sample Form (Powder/Film/Others)	POWDER		
Sample-Electron beam interaction	☐ Conductive ☑ Non-conductive	☐ Conductive ☐ Non-conductive	☐ Conductive ☐ Non-conductive
Coating			

Additional Relevant Sample Information (Sample Preparation, Storage conditions, Hazard Information)

THANK YOU FOR YOUR BUSINESS!

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