

**REMEDICATION OF HOME-TREATED WATER BY REMOVAL OF
RESIDUAL ALUMINIUM AND PHENOLIC COMPOUNDS USING GREEN
SYNTHESIZED MAGHEMITE ($\gamma\text{Fe}_2\text{O}_3$) NANO PARTICLES**

Daniel Murage

**A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of
Doctor of Philosophy in Chemistry of Murang'a University of Technology**

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DECLARATION

Student declaration

I hereby declare that this thesis is my original work and, to the best of my knowledge, has not been presented with a degree award at this university or any other university. I understand what plagiarism is and I am aware of the university's policy on this. I understand that any false claim regarding of this work shall result in disciplinary action in accordance with the University Anti-Plagiarism Policy.

Daniel Murage

Date

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Supervisors' Approval

The undersigned certify that they have read and hereby recommended for acceptance of Murang'a University of Technology a thesis entitled **“Remediation of Home-Treated Water by Removal of Residual Aluminium and Phenolic Compounds Using Green Synthesized Maghemite ($\gamma\text{Fe}_2\text{O}_3$) Nano Particles”**.

Prof. Sylvia Opiyo,

Date

Department of Physical and Biological Sciences,
Murang'a University of Technology.

Prof. Peter Waithaka,

Date

Department of Physical and Biological Sciences,
Murang'a University of Technology.

Dr. Sharon Kiprotich,

Date

Department of Physical and Biological Sciences,
Murang'a University of Technology.

DEDICATION

I dedicate this document to my children Olive Wanjia, Clarence Karuma and Adrian Murage. To my lovely wife Grace Wanjiru who has been a strong pillar in my life. Also, to my father Mr. Elias Murage Muya, who has been a strong source of advice, support and more so encouragement. To my siblings, Esther Njeri, David Ng'ang'a, Jeremiah Muhoro, Mary Wambui, Faith Wanjia and Jese Maara not forgetting Rahab, Hannah, Mr. Kanyi, Bishop Ben and Dr Njuguna for your prayers and support. Dr John Njuguna you specifically advised me to do post graduate studies in chemistry. My friends and workmates Madam Winrose, Joseph Irungu, Muchoki, Mercy Mwaniki, Mirriam Wanjohi, Catherine Maina, Harun Bosuben and Anastasia Kithinji, may God bless you all including those not mentioned.

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ABSTRACT

Water is an essential natural resource for support of life. To make water safe for human consumption, water needs to be treated. However, small-scale consumers, use home water treatment coagulants obtained from commercial shops. Example of coagulants include 'Rock Alum' available in Kenyan retail shops. The use of aluminium based coagulants leaves residual aluminium in water. Aluminium exposure through contaminated water results in neurological disorders. Phenolic compounds occur naturally in water mainly from plants and environment. Their presence in drinking water causes muscles weakness and respiratory disorders. This study explored the preparation of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticle by a green method using pineapple (*Ananas comosus*) peels extract and their application in remediation of treated water by removal of residual aluminium ions and phenolic compounds. The nanoparticles were characterized using X-Ray Diffraction (XRD), Ultra-Violet visible light Spectrometer (UV-Vis) and Fourier Transform Infra-Red spectrometer (FTIR). The maghemite nanoparticles (0.5 g) were used in batch adsorption of residual aluminium ions and phenolic compounds from water samples at varying pH, contact time and temperature. The de-ionized water samples treated with 'Rock Alum' coagulants gave residual aluminium ions at concentration of 1.9 ppm when 0.0125 g of coagulant (Rock Alum) was used. This concentration of aluminium ions was higher than permissible limits by the World Health Organization of 0.1 ppm. On characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using XRD, FTIR and UV-Vis, it was found that at high calcinating temperature of 400-450 °C gave nanoparticles with improved crystallinity and enhanced ordering of the maghemite structure. The percentage adsorption of aluminium ions was determined using Atomic Absorption Spectrometer (AAS) and UV-Vis for phenolic compounds. It was found that adsorption decreases with increase in pH for aluminium ions. Temperature had little effect on adsorption, while rate of adsorption increased with increase in contact time. Adsorption of aluminium was multilayer and fitted to Freundlich adsorption isotherm. Maghemite was found to be efficient in adsorption of phenolic compounds (Phenol and resorcinol) from spiked de-ionized water samples. Phenol was best adsorbed at pH of 5 and 7 for resorcinol. Rate of adsorption decreased slightly in both phenol (3.6%) and resorcinol (15.2%) with increase in adsorption temperature. At optimum adsorption conditions pH, temperature and contact time, mean adsorption of aluminium, phenol and resorcinol was 75.63, 72.61 and 80.09% respectively. This study recommends use of alternative water coagulants which are not aluminium-based.

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ACRONYMS AND ABBREVIATIONS

AC	Activated Carbon
AAS	Atomic Absorption Spectroscopy
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
CNTs	Carbon Nano Tubes
FTIR	Fourier Transform Infra-Red Spectrophotometer
FWHM	Full Width at Half Maximum
GC-MS	Gas Chromatography-Mass Spectrometer
IARC	International Agency for Research on Cancer.
KEBS	Kenya Bureau of Standards
SEM	Scanning Electron Microscope
TSS	Total Suspended Solids
UNESCO	United Nations Educational, Scientific and Cultural Organization
UV-VIS	Ultra-Violet Visible Spectroscopy
WHO	World Health Organization
WTP	Water Treatment Plants
WTR	Water Treatment Residuals
XRD	X-Ray Diffraction

CHAPTER ONE INTRODUCTION

1.1 Background of the Study

Water is an essential natural resource for human existence. About 70% of the surface of the earth is covered by water. However, less than 4% of the available water is fit for human consumption (Mishra, 2023). Demand for clean and safe water has greatly increased with the increase in human population and industrial developments. In Africa, about 80% of the residents use natural water sources such as rivers, streams, wells, and lakes for their domestic uses. Majority of these natural sources provide untreated water. Raw water may contain physical debris, biological microorganisms and other chemical contaminants. Water gets contaminated as result of agricultural activities such as farm chemicals, and fertilizers (Okafor *et al.*, 2024). In addition, water contamination may also arise from landfills, illegal garbage disposal and industrial processes especially industrial water discharges to natural water sources such as rivers and streams.

Water treatment is fundamental to ensure the safety of consumers (Samburskiy and Grodzenskiy, 2020). Raw water from surface sources such as rivers, streams, ponds, freshwater lakes, and many others is usually unsafe for human consumption. Therefore, it requires quality correction to make it safe. In water treatment processes, suspended solids, organic matter, chemical contaminants, and bacteria are removed. The water treatment process has various stages which involves coagulation and flocculation of suspended particles. Other stages include, sedimentation, filtration, and disinfection to make water safe. In water treatment, coagulants such as aluminium sulphate, poly aluminium chloride, and iron salts are commonly used. Coagulants residuals are removed from water in the form of flocs. These are macro-particles that are further removed by filtration and sedimentation.

In Kenya and many other African countries, only about 35% of the population have access to treated tap water provided by county governments. The need for clean water makes local communities carry out domestic water treatment of the water obtained from local streams, rivers, wells, or boreholes. Home water treatment involves the use of commercially available water treatment coagulants.

Water treated at home using commercial coagulants does not undergo quality checks by government bodies such as the Kenya Bureau of Standards (KEBS) unlike in the case of public water treatment plants. This may result in the presence of harmful Water Treatment Residuals (WTR) in home-treated water endangering the health of the consumers. Some of the home water treatment coagulants added to the raw water remain in the water after flocculation and filtration resulting in water treatment residuals (WTR) such as aluminium and phenolic compounds. In addition, water also contains some metallic ions signature of the water source.

Contaminated water with residual metal ions may be remediated by various methods which includes reverse osmosis, precipitation using chemicals, electrodialysis, ion exchange, and adsorption using nanoparticles. Nanotechnology is a modern science that deals with the use of materials whose particles size are at a nanoscale of a billionth of a metre. Due to their small size, nanoparticles have a very large surface area. This provides sufficient area for function groups and ions to bond. In addition, the surface area of nanoparticles is easily and conveniently modified. Therefore, nanoparticles are an efficient method of removing metal ions from water.

Home-treated water is believed to have residual metal ions, especially aluminium ions. This is because after coagulation some metal ions remain in water as well as phenolic groups that are present in most water sourced from surface sources such as streams, rivers, and lakes as well as shallow wells. Human exposure to high levels aluminium for a long period may suffer from long lasting effects of aluminium poisoning. Aluminium poisoning beyond the tolerable limits of the human body, is linked to various neurological disorders like multiple sclerosis and Parkinson's disease (Farhat & Ali, 2020).

Nanoscale materials are an effective method of water remediation. To mention a few, these materials include manganese oxide, zinc oxide, titanium oxide, graphene, carbon nanotubes, zero-valent metals, and iron oxides such as magnetite, hematite and maghemite. Iron oxide nanoparticles are effective adsorbents of metal ions in water due to their porous surface. They adsorb by forming ionic, covalent, and in some cases metallic bonds with the adsorbate ions (EthaiB *et al.*, 2022). Adsorption columns packed with iron-based nanomaterials have been applied on a large scale in the

remediation of water contaminated with metal ions such as Arsenic, lead, manganese, chromium, nickel, copper, and many others.

Previous studies have reported that maghemite ($\gamma\text{Fe}_2\text{O}_3$) is an efficient adsorbent which can be applied in removal of metal ions from contaminated water. It's effectiveness in water remediation is attributed to its small particle sizes of less than 9 nm resulting in large surface area and the magnetic properties. Maghemite can be prepared by chemical precipitation or by green synthesis among other methods.

Green synthesis of nanoparticles involves use of extracts from plant materials and other biological substances as oxidizing or capping agents in various reactions in chemical preparation of nanoparticles. Plant extracts contain antioxidants such as polyphenols, phenolic acids, and flavonoids among other bio-chemical compounds which have strong reducing properties. Previous studies have reported use of plant extracts in preparation of nanoparticles by precipitation from their salts. This study employs use of plant extract prepared from pineapple (*Ananas comosus*) peels which are common organic wastes from most local markets especially in central and coastal Kenyan regions.

Variations in synthesis conditions have a great effect on the properties of the nanoparticles obtained such as particle size. Change in calcination temperature is believed to have an effect on the characteristics of the nanoparticles formed, therefore in this study temperature was varied. The rate of adsorption of metal ions from water is affected by various parameters such as pH, contact time, adsorbent dosage, and operating temperature (Sosun *et al.*, 2022).

1.2 Problem Statement

Water treatment is a key safety requirement in the provision of water for human consumption and domestic uses. Aluminium-based coagulants are the most common coagulants used in home water treatment. However, they form by-products such as aluminium ion residues and other compounds which remain in the water after the flocculation process. Higher concentrations of aluminium in treated water beyond $300 \mu\text{g}/\text{dm}^3$ may endanger human health. Home-treated water, it has been reported to contain different phenolic compounds especially if it originates from surface and underground sources. Aluminium residues and phenols have detrimental effects on

human health. Ingestion of aluminium is associated with neurological disorders which includes Alzheimer's disease and dementia to the victims (Batoool *et al.*, 2021; Murage & Waithaka, 2025).

Water treatment using chlorine-based compounds and other water disinfectants is linked with the formation of different residual phenolic compounds. Human consumption of water-containing phenolic compounds is associated with several health challenges which include muscle weakness, irregular breathing, respiratory disorders, and impaired coordination (Bryliński *et al.*, 2023). The compounds have high mutagenic and therefore, could be carcinogenic (Niu, 2023). Phenolic compounds contaminants also contribute to bad taste and odour in water (Ahmaruzzaman *et al.*, 2024).

In previous research, aimed at finding a convenient remediation technique for contaminated water, iron oxide nanoparticles were synthesized using various methods such as co-precipitation, sol-gel, and irradiation. Most of these chemical processes involve the use of toxic compounds (Ekwumengbo *et al.*, 2023). This study investigated the use of green synthesis of maghemite nanoparticles. This method is environmentally friendly and do not generate hazardous by-products.

Exposure to aluminium ions and phenolic compounds is linked to many health challenges for humans. Therefore, remediation of treated water by eliminating these metallic ion and phenol compound residues after water treatment is of great importance. Most traditional remediation techniques such as membrane filtration, ion exchange, and reverse osmosis are expensive to regenerate, have high energy requirements, and produce toxic wastes. Therefore, the use of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles in water remediation provides an effective and convenient method to address this challenge. In addition, their low cost and efficient adsorption capacity are some of their advantages (Samburskiy and Grodzenskiy, 2020; Ethaib *et al.*, 2022; Mishra, 2023). This study focused on investigating the content levels of residual aluminium ions in the treated water and adsorption of residual aluminium ions and phenolic compounds.

1.3 Objectives of the Study

1.3.1 General Objectives

To investigate the removal of residual aluminium and phenolic compounds in water samples treated with home water treatment coagulant (Rock Alum) and the remediation of the same using maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles prepared by green synthesis.

1.3.2 Specific Objectives

- i. To determine concentration levels of residual aluminium in de-ionized and natural water samples treated using home water treatment coagulant (Rock Alum).
- ii. To synthesis and characterize maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles from iron (iii) chloride by green synthesis using pineapple (*Ananas comosus*) peels extract as capping agent at different calcination temperatures.
- iii. To determine optimum conditions of pH, temperature and contact time for adsorption of residual aluminium from de-ionized and natural (River and borehole) water samples treated using home water treatment coagulant (Rock Alum).
- iv. To determine optimum conditions of pH, temperature and contact time for adsorption of phenolic compounds (phenol and resorcinol) from spiked de-ionized water.
- v. To determine the effectiveness of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles in the removal of both aluminium and phenolic compounds from water samples at optimum conditions.

1.4 Research Questions

- i. Does water treated using home water treatment coagulants (Rock Alum) contain residual aluminium ions which are above the limits of 0.1 ppm set by World Health Organization (WHO)?
- ii. How does change in calcination temperature influences the physical characteristics of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles synthesized from iron (iii) chloride using pineapple (*Ananas comosus*) peel extract as capping agent?
- iii. How does the change in conditions of pH, temperature and contact time affect adsorption of residual aluminium from de-ionized and natural water samples

treated using home water treatment coagulant (Rock Alum) using maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticle adsorbent?

- iv. How does change in conditions of pH, temperature and contact time affect adsorption of phenolic compounds from spiked de-ionized water using maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticle adsorbent?
- v. How effective is maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles in adsorption of both residual aluminium and phenolic compounds from water at optimum conditions?

1.5 The Significance of the Study

This study will assist the National government through the Ministry of Health, County governments to appreciate effects of use of home water treatment coagulants in water treatment. It will also enlighten the chemical industries that manufacture and sell home water treatment coagulants (Rock Alum), on need to improve their products to reduce residual aluminium in treated water. This knowledge is important to understand and apply modern domestic water treatment techniques. Most common water treatment techniques have a wide range of limitations and drawbacks that may be harmful to humans. This research will be of great importance in addressing these drawbacks efficiently and cheaply.

Aluminium salts are one of the most commonly used compounds in water treatment. Traditional flocculation may not eliminate all aluminium residues. Consumption of contaminated water results in aluminium ion poisoning. Aluminium exposure is well documented by previous researchers and its negative effects on human health. This research will be important in solving this problem by introducing new knowledge on the removal of residual aluminium ions using maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticle adsorbents. This study also explores cheaper and efficient method of nanoparticle synthesis by green method.

This research will enlighten governmental organizations on the need for carrying out regular research to keep up with finding solutions to emerging challenges related to water treatment. This work also stirs interest in researchers in the field application of adsorbents in addressing problems of water pollution due to the presence of metallic ions in water, hence can be used in wastewater recycling. This is important knowledge

that Governmental and Non-Governmental Organizations can use when addressing the problem of water scarcity in Kenya and the world.

1.6 Scope of the Study

This study focused on the adsorption of aluminium and phenolic compounds (phenol and resorcinol) from spiked water samples treated by home water treatment coagulants (Rock Alum). Adsorption survey was done using maghemite prepared by the green method only. The adsorption was carried out at different pH levels ranging from 3, 4, 5, 6, 7, 8 and 9. At varying temperature of 35, 45, 55, 65 and 75 °C. At different contact time of 15, 30, 45, 60 to 75 minutes. In adsorption of phenolic compounds from spiked de-ionized water at varying contact time of 5, 10, 15, 20, 25, and 30 minutes, varying temperature of 20, 30, 40, 50, 60 and 70 °C and pH range of 3, 5, 7, 9 and 11.

1.7 Limitations of the Study

In this study, the research focused on synthesis of maghemite nanoparticles by green synthesis using pineapple (*Ananas comosus*) as the capping agent in reduction of iron (iii) chloride without studying other methods of synthesis of nanoparticles. De-ionized water was obtained from the laboratory, natural water samples collected from three rivers; Chania, Thika and Maragwa river. Borehole water samples collected from three areas; Juja, Thika and Maragwa regions were also used in the study. The adsorption was done from aluminium ions only while only phenol and resorcinol were adsorbed from spiked de-ionized water samples.

1.8 Contributions of the Thesis

This study enlightens on effect of use of aluminium based coagulants (Rock Alum) in treatment of water. This study will contribute to the body of knowledge by appreciate efficient and cost method of synthesizing maghemite nanoparticles. It investigates and reports on the factors affecting the rate of adsorption of aluminium and phenolic compounds from water.

1.9 Organization of the Thesis

This thesis has been arranged into five chapters. Chapter one presents the background of the water treatment, residual aluminium and occurrence of phenolic compounds in water and the role of maghemite nanoparticles in water remediation. It also presents in the general, specific objectives as well as research questions and significance of

study. The second chapter discusses in details water contamination and treatment procedure. It also discusses the occurrence of aluminium and phenolic compounds in water and their effects. Synthesis and characterization of maghemite nanoparticles is also discussed bring out the research gap. Chapter three presents experimental procedures used as per specific objective. Chapter four presents the results and discussion on determination of residual aluminium in water treated using home water treatment coagulant. It also presents results on characterization of maghemite nanoparticles. Results on adsorption of aluminium and phenolic compounds at optimized conditions of pH, contact time and temperature are also presented. Chapter five summarizes the results and offers recommendations from the study.

CHAPTER TWO LITERATURE REVIEW

2.1 Introduction

In this chapter, detailed research findings by other researchers related to this study are discussed. This discussion includes the specific area of study, methods used, results obtained and conclusions made.

2.2 Water contamination

Globally, man is confronted with two major challenges which are scarcity of clean water and water pollution (Oladoye, 2022). In Africa drinking water is commonly obtained from natural sources such as rivers, streams, boreholes, and lakes (Bazaanah & Mothapo, 2023; Lapworth *et al.*, 2020). A study by (Okafor *et al.*, 2024), reported that globally, about half of the population do not have access to clean and safe drinking water at least some seasons of the year (Santa-Cruz-Mérida *et al.*, 2025). Currently, there are many health challenges affecting a large human population globally which are attributed to domestic use of raw (untreated) water bringing the need for efficient water treatment (Murage *et al.*, 2022).

Raw water from most of natural sources such as rivers, streams, boreholes and lakes tends to be contaminated by metal ions, phenolic groups, soluble salts, bacteria, suspended particles, and soil (Fahmy *et al.*, 2023). High levels of inorganic nutrients in water accelerates growth of aquatic plants such as algal blooms depleting dissolved oxygen in the water. This kills aquatic life due to lack of oxygen also causing water body to stink (Tariq & Mushtaq, 2023). In addition, high levels of water contaminants contribute to increases in levels of Total Dissolved Solids (TDS) and Biological Oxygen Demand (BOD). Other water parameters affected by water pollution include, Total Suspended Solids (TSS) and Chemical Oxygen Demand, (COD). Untreated natural water is also known to contain other contaminants such as inorganic pollutants, oil spillage, water soluble radio-active substances, heavy metal ions among other pollutants especially from industrial and agricultural wastes (Tariq & Mushtaq, 2023). These pollutants negatively affect the health of those who use untreated water for their domestic uses (Gupta & Gupta, 2020; Qasem *et al.*, 2021).

Consumption of untreated water may result in health challenges like water-borne pathogens such as blood flukes and nematodes. Untreated water transmits waterborne diseases such as gastrointestinal infections, protozoal diarrhea, bacterial pneumonia, and tissue helminths (Ali *et al.*, n.d.; Bazaanah & Mothapo, 2023). In a study on water treatment, (Tariq & Mushtaq, 2023) reported that about 80% of communicable diseases are transmitted through use of untreated water. Contamination of water from untreated wastewater contributes to transmission of infectious diseases such as typhoid, cholera, diarrhea, fever, vomiting, skin and kidney problems (Thatai *et al.*, 2018). These diseases may be in epidemic and endemic forms which causes morbidity and mortality, especially in children and also adults as observed in the poorest Democratic republic of Congo, Afghanistan and India (Tariq & Mushtaq, 2023; Thatai *et al.*, 2018). The risks associated with contaminated water presents the need for water treatment to make it safe for human consumption.

2.3 Conventional water treatment methods

Water treatment processes are used to prepare safe and clean water ready for human consumption from raw water. In the past rudimentary methods of water treatment such as filtration using clothes, decantation and boiling were commonly used (Samburskiy & Grodzenskiy, 2020). However, with advancement in technology, more efficient techniques are in use in modern times. Some of most recent methods includes reverse osmosis, ultraviolet disinfection, nano-filtration among others. Raw water is passed through various stages of treatment where different contaminants are eliminated. In general water treatment involves many units which include settling, coagulation, flocculation, sedimentation, filtration, and disinfection (Abd Nasier & Abdulrazzaq, 2021; Pillai & Thombre, 2024).

2.3.1 Screening

This stage involves removal of large solid materials such as wooden logs, leaves, plastics papers, small animals and any other materials that might be suspended in water by filtration at the water catchment point. This is done by passing water through physical barriers whose holes vary in size depending on the size of target debris (Fahmy *et al.*, 2023).

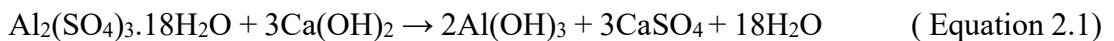
2.3.2 Settling

During the settling stage, raw water is stored in large tanks to facilitate the settling of large suspended particles and other solid materials such as sand and small stones. When large particles are removed, water is transferred to a coagulation tank (Thatai *et al.*, 2018; Lapworth *et al.*, 2020; Abd Nasier & Abdulrazzaq, 2021).

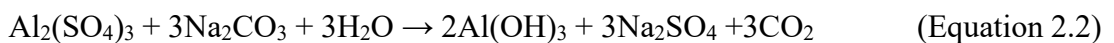
2.3.3 Coagulation

Coagulation is a chemical process is carried out to enhance sedimentation. At the coagulation tank, coagulants are added to the water. Coagulants are chemical compounds consisting of cations. These ions bonds and cancel out the negative charge of dirt and other suspended particles in the water. Once the charges are neutralized the suspended particles bind together forming macro particles. Aluminium and iron salts are the most commonly used Coagulants (Pillai & Thombre, 2024; Smotraiev *et al.*, 2022). Aluminium sulphate ($\text{Al}_2[\text{SO}_4]_3 \cdot 14\text{H}_2\text{O}$) is the most commonly used for this purpose. Other Coagulants include, iron(iii)sulphate ($\text{Fe}_2[\text{SO}_4]_3$), Iron (iii) chloride (FeCl_3), magnesium carbonate (MgCO_3) and slaked lime ($\text{Ca}[\text{OH}]_2$). In some cases, sodium aluminate (NaAlO_2) is also used as a coagulant. Recently, the use of polyaluminium chlorides (PACl). This has been observed in some countries (Krupińska, 2020; Bagastyo *et al.*, 2023).

During coagulation, aluminium sulphate reacts with hydrated lime forming flocs of aluminium hydroxide as shown by Equation 2.1



When water has been treated with a low alkaline character, alkaline compounds such as sodium carbonate, sodium hydroxide, or hydrated lime are added. Sodium carbonate (soda ash) is the most commonly used buffering agent as shown by Equation 2.2.



Aluminium cations (Al^{+3}) form Van Der Waals bonds with the colloid matter in water resulting in the formation of micro-flocs. The repeated binding with particulate,

colloidal, and dissolved substances occur forming macro molecules (Mohamed *et al.*, 2020; Pillai & Thombre, 2024).

2.3.4 Flocculation

During flocculation process, water is gently mixed with flocculant chemicals. This results in formation of relatively bigger and heavier particles known as flocs. The size of the flocs is usually increased by constant gentle agitation. This can increase the size of flocs from submicroscopic size to visible suspended particles. The continuous mixing increases collisions and interactions with coagulants resulting in increase of size of the flocs (Pillai & Thombre, 2024). Once the flocs have achieved the optimum size, the water is ready for sedimentation. The success of flocculation depends on the completeness of the coagulation stages (Bagastyo *et al.*, 2023; Smotraiev *et al.*, 2022; Tahraoui *et al.*, 2024).

2.3.5 Sedimentation

The treated water sample is stored for several hours to allow the flocs formed in the coagulation and flocculation stages to settle at the floor of the tank. The accumulated solid materials at the bottom of the sedimentation tank are known as sludge (Mgbechi, 2025; Qrenawi & Rabah, 2021; Rai, 2024). The sludge consists of organic and inorganic residues from the water source and also added treatment chemicals. The sludge may also contain residues sourced from other human related activities such as mining, farming and industrial effluents along the water path (Tahraoui *et al.*, 2024). The water source and treatment process affects the characteristics of the final sludge. In commercial water treatment process, the sludge is usually dried and landfilled (Pillai & Thombre, 2024).

2.3.6 Filtration

Treated water is passed through filters to remove the solid particles from the water. These filters consist of gravel, sand, or charcoal. Filtration using activated charcoal is also effective in removing bad odours. Filters are cleaned regularly by backwashing (Elmogy, 2023). Ultra-filtration is sometimes applied where water is passed through filter membranes with very small pores. This allows only water to pass through while small molecules such as salts and charged particles are removed (Attah *et al.*, 2023; Elmogy, 2023).

2.3.7 Disinfection

Water disinfection is done to killing or inactivating parasites, viruses, bacteria, and other disease-causing microorganisms. This is done to ensure water is safe including killing harmful micro-organisms that may be in the distribution line (Demirel, n.d.; Ghernaout & Elboughdiri, 2020).

In water treatment disinfection can be in form of physical or chemical disinfection. In physical disinfection it involves use of ultra-violet radiation while chemical disinfection involves use of chloramine, ozone, chlorine and dioxides. Ultra-violet radiation is commonly applied in disinfection of effluents. It has advantage of not giving harmful by-products which might reduce water quality. UV radiation disables the growth and replication of micro-organism by destroying their deoxyribonucleic acid. This method is safe since no chemical reagent is added to water during disinfection process (Kumar, 2021; Tahraoui *et al.*, 2024).

Chloroamination as method of disinfection involves reacting chlorine and ammonia under controlled conditions forming mono-chloramine. This is done to prevent formation of by-products which affects the taste of water. Chlorination is the most common method of water disinfection. It involves addition of chlorine into water. When chlorine is added to water, it reacts to form hypochlorous and hypochlorite ions which are strong oxidizing agents. Chlorination or addition of chlorine-based disinfectants are cheap method however they chloride residues therefore post-treatment is necessary (Newman, 2021; Stawarz-Janeczek *et al.*, 2021).

2.4 Home water treatment

In water treatment, different chemical compounds are added in to water to coagulate suspended solid and dissolved impurities in water. The precipitated flocs sink at the bottom due to their increased density. Globally, Alum [$\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$], ferric chloride [$\text{FeCl}_3 \cdot \text{H}_2\text{O}$] and polyelectrolytes such as poly aluminium chloride, are commonly used coagulants (Mohamed *et al.*, 2020). In Kenya, many citizens are not served with clean water from commercial water treatment plants. This has brought the need for the use of home water treatment coagulants (Rock Alum) in the treatment of water from local sources mainly borehole water or from streams or rivers. In the use of these coagulants, the consumer follows the instructions as directed by the manufacturer. In most cases, the instructions involve the addition of a given mass of

coagulant in a given volume of water. In these instructions, the procedure for filtration of the flocs and remediation techniques for removal of residual aluminium from water are not included. These coagulants include brands such as “Rock Alum” (Trus *et al.*, 2023; Tuan Vo *et al.*, 2020).

2.5 Residual aluminium in treated water

Water treatment in conventional water treatment plants as well as in-home treatment involves the use of aluminium or iron-based coagulants. Specifically, aluminium sulphate, sodium aluminate, and iron (ii) sulphate salts are most common commercial coagulants used in water treatment (Fahmy *et al.*, 2023). Aluminum sulphate after is added in to raw water, hydrolysis occurs and within a pH range of between 6.7 to 7.3 resulting in coagulation. At pH below or above the range, aluminium sulphate does not coagulate resulting in remaining residual ions in water (Yuan *et al.*, 2022). Sometimes residual aluminium and iron (ii) ions in treated water could be in both dissolved and in precipitated complex forms. It has been observed that acid rain has elevated residual aluminium in treated water due to low pH which dissolves aluminium salts in rocks and soils increasing residual aluminium (Krupińska, 2020).

Previous studies have reported that water treated with aluminium salts has elevated residual aluminium content than raw water itself. In most cases, the type of salt used in coagulation process determines the residual metals in the treated water (Krupińska, 2020; Wallace *et al.*, 2023). A study done in Indian villages, Suja George (2010) found that alumina-treated groundwater at around neutral pH contained residual aluminum concentration of 0.1-0.3 mg/L which level was higher than acceptable limit by WHO (George *et al.*, 2010; Lapworth *et al.*, 2020).

It has also been observed that residual metal ions in treated water can be caused by the mineral and elemental signature of the source water (Zhao *et al.*, 2025). According to the World Health Organization (WHO) regulation 1997, residual aluminium ions in water are a maximum of 0.2mg/L. However, most government guarantee the safety of its citizens by advocating for tolerable threshold limit of 0.1 mg/L of residual aluminium in drinking water. Presence of high concentrations residual aluminium ions in treated water sometimes out ways the benefits of use of “Alum” in coagulation (Krupińska, 2020; Yuan *et al.*, 2022).

2.6 Aluminium ions in water

Across the globe, clean and safe water is not available to about 40% residents especially in developing countries. In absence of clean water, the population use water from natural sources for domestic purposes. These sources include, streams, ponds, underground wells, rivers and lakes. Tremendous growth in global population has greatly contributed to rapid urbanization. This has created more demand for safe and clean water as population increases in almost every part of the world. Anthropogenic activities such as agriculture and industrial development are major contributors to water pollution. Large quantities of water are used in most industries. Some industries discharge effluents into water sources. These discharges may contain toxic metallic pollutants such as aluminium, lead, chromium, silver and copper (Das & Poater, 2021; Qasem *et al.*, 2021). These discharges contain toxic substances dissolved in the water. Their leaching into the soil also contaminates surface and underground water sources. Domestic use of such contaminated water contributes to aluminium poisoning to the population (Ahmed *et al.*, 2021; Zhao *et al.*, 2025).

Previous studies have reported that untreated water from natural sources may contain high concentrations of aluminium at range of 0.02-0.4 ppm among other metallic ions. This observation was explained to be as a result of surface and underground waters interacting with soils and rocks containing aluminium salts such as bentonite and zeolite (Farhat & Ali, 2020; Niu, 2023). In a study about aluminium content in groundwater in the southern part of Pakistan, (Azizullah *et al.*, 2021) reported that water contained elevated levels of aluminium ions. Water was found to contain between 0.95-1.92 ppm which is beyond permissible levels of 0.2 ppm by the Environmental Protection Agency (EPA). This was attributed to water passing through soil and rocks as well as industrial pollution (Azizullah *et al.*, 2021).

Acid rain is also known to contribute greatly to elevated aluminium levels in many natural water sources as a result of reactions of aluminum-containing rocks in acidic rain (Niu, 2023). Aluminium exists as Al^{+3} which is an ion with strong polarizing properties. Therefore, its solubility in water is high in an acidic or alkaline environment ($6 < pH < 8.5$). Consumption of water containing elevated amounts of residual aluminium in water has a huge effect on human health. This has contributed to various researchers advising against treatment of drinking water using of

aluminium-based coagulants (Farhat & Ali, 2020; Petrunin *et al.*, 2023). Aluminium poisoning can be caused by domestic use of these waters.

2.7 Effects of mild aluminium exposure on human health

Aluminum uptake through the gastrointestinal tract occurs in proportion of between 0.1 to 0.3 %. This absorption occurs at upper intestines. The elevated absorption is attributed to its lower pH (Krause, 2021). Niu (2023) reported that gastrointestinal uptake to be between 0.1 and 0.4% from water. However, aluminium absorption into human body sometimes can be higher to about 5% especially from medical drugs (Jiang *et al.*, 2025; Niu, 2023; Schönenberger *et al.*, 2025). Aluminum absorbed in to the human body is mainly excreted through sweating, semen, skin, nails, sebum, faeces and urine (Affourtit *et al.*, 2020; Murage & Waithaka, 2025). A healthy adult with well-functioning kidneys is able to efficiently eliminate most of aluminium through urine. Therefore, most adults can cope with the daily environmental exposure to aluminium. However, according to Alabi *et al.*, (2020), removal of aluminium through urine does not prevent risks of its accumulation in other parts of the body.

Mild exposure of aluminium results in general symptoms such as fatigue, confusion, dizziness and somnolence. Other symptoms include abdominal pains, nausea, vomiting sometimes diarrhea. When a victim is exposed to aluminium fumes, they may experience persistent coughing and difficulty breathing (Jackson & Rout, 2024).

2.8 Effects of acute aluminium exposure on human health

A person exposed to high levels aluminium for a long period may suffer from long lasting effects of aluminium poisoning. This is as a result of bioaccumulation of aluminium in body organs. In most cases metals accumulates in liver, kidney, brains and sometimes bones (Coulson & Hughes, 2022). This results in long term effects on the health of the victim. These effects are more pronounced in young children and in people with renal problems due to their reduced renal excretion of aluminium (Affourtit *et al.*, 2020; Coulson & Hughes, 2022; Jackson & Rout, 2024). The effects of acute aluminium exposure on human health include the following:

2.8.1 Effects on central nervous system

Various researchers have reported that exposure to high levels of aluminium for an extended period of time is harmful to human health. Aluminium poisoning has direct

effect on central nervous, bones and hematopoietic systems (Dey & Singh, 2022). Effects of exposure to aluminium targets central nervous system. Human exposure to aluminium causes several neurological problems the main one been aluminium encephalopathy. This condition exhibits challenges such as cognitive impairment, mental confusion, agitation, and myoclonic jerks. In extreme case, it may result in seizures, coma and death. Other neurological consequences include speech difficulties, dementia and depressive mood changes (Rahimzadeh *et al.*, 2022).

Other studies found that exposure to aluminium has neurological effects ranging from disruption of neuro-transmitters, neuro-inflammations, cytoskeletal dysregulation among other effects. These defects are as a result of accumulation of aluminium which causes increase in biosynthesis of transferrin receptors (Jackson & Rout, 2024). It has been reported that neurological disorders which include multiple sclerosis, diminished intellectual function and forgetfulness are directly linked to exposure to aluminium beyond the tolerable limits (Affourtit *et al.*, 2020; Farhat & Ali, 2020).

2.8.2 Alzheimer's Disease

Alzheimer's disease (AD) is characterized by disruption in perception and mis-interpretation of time. In addition, disorientation, memory problems, language abilities and compromised intellectual ability may also manifested. Alzheimer's disease causes cerebral dysfunctional which leads to difficulties in reception and processing of a new information signal reducing cognitive and motor functions (Anwal, 2021; Ferrari & Sorbi, 2021; Polsinelli & Apostolova, 2022). Human exposure to aluminium beyond tolerable limits is believed to be one of the main causes of Alzheimer's disease. In addition, other related neurological disorders such as dementia and impaired speech can be caused by aluminium exposure. Aluminium poisoning through consumption of water may cause Alzheimer's disease and related disorders to healthy person (Coulson & Hughes, 2022; Murage & Waithaka, 2025). Previous study on aluminium exposure had reported that exposure to high concentrations of aluminium ions through water causes acute signs of dementia and sometimes Parkinson's disease especially in people with kidney failure (Bryliński *et al.*, 2023; Dey & Singh, 2022; Niu, 2023).

2.8.3 Parkinson's Disease

Most common effect attributed to aluminium poisoning after Alzheimer's disease is Parkinson's disease. It is also a neurodegenerative disease indicated by selective death

of nerve cells. The death is a result of several disorders such as oxidative stress, protein quality disorder, microglia activation and mitochondrial dysfunction among others. These conditions are as a result of accumulation of aluminium and iron(iii) ions (Raj *et al.*, 2021; Vrbanc *et al.*, 2024).

2.8.4 Renal Failure

Aluminium is primarily excreted from human body through kidneys therefore renal functions are more affected by the aluminium exposure. Effects of aluminium poisoning are more severe in patients with other renal disorders. This results in kidney failure due to disorders such as nephrotic syndrome, acute renal glomerulonephritis, disrupts renal tubular transport which affects sodium/potassium-water balance (Jackson & Rout, 2024). In addition, Hîncu *et al* (2024) reported that patients with intestinal diseases have high likelihood of suffering from high bioaccumulation of aluminium in their bones. This observation was also made in patients undergoing dialysis for chronic kidney failure, and patients on long term prescription of antacids. Oral consumption of aluminium-based antacids for an extended period of time contributes greatly to aluminium poisoning. However, the rate of absorption depends on the chemical form of the medication used (Hîncu *et al.*, 2024; Murage & Waithaka, 2025; Niu, 2023).

2.8.5 Bone defects

Absorption of aluminium interferes with absorption of calcium in a process known as bone demineralization by calcium replacement. This can affect bone formation. In addition, accumulation of aluminium in bones results in reduced differentiation of osteoblasts, bones defect and increased the risk of bone-related disorders. These challenges include conditions such as osteomalacia and osteoporosis. Bone defects are more severe to children who are in the early bone developmental stage (Gökmen & Gül, 2023; Jackson & Rout, 2024).

2.8.6 Reproductive disorders

Aluminium exposure may have negative effects on the reproductive health of the victims. Jackson and Rout (2024) reported that, there is potential link between aluminium accumulation in semen with fertility challenges. A study on rats exposed to aluminium for 60 days revealed a decrease in sperm count, reduced number of

normal sperms as well as poor sperm mobility. This demonstrated effect of aluminium poisoning on reproductive health (Aghashahi *et al.*, 2020; Maghraoui *et al.*, 2023).

2.8.7 Pulmonary effects

Exposure to aluminium dust results in bronchial hyperresponsiveness. This condition is reversible especially in mild fumes. This is characterized by shortness of breath, dyspnea and coughs. These challenges are common to workers who work in aluminium milling plants, electrolytic plants etc. in extreme cases pulmonary tissue damage can occur resulting various pulmonary disorders such as asthma, chronic pneumonia, pulmonary fibrosis chronic bronchitis among others (Gökmen & Gül, 2023; Jackson & Rout, 2024).

2.9 Phenolic compounds

Phenols and their compounds are aromatic environmental pollutants that are highly toxic to humans. Phenolic compounds consist of benzene ring with one or more hydroxyl groups attached. For example, phenol have a single hydroxy attached, while catechol and resorcinol have two hydroxyl groups attached (Ramos *et al.*, 2024). Phenolic compounds can be classified broadly as halogenated or non-halogenated. Examples of halogenated phenolic compounds include 2-chlorophenol and 2,4-dichlorophenol. While 2,4-dinitrophenol and 4-methylphenol are examples of non-halogenated phenols (Ndwabu *et al.*, 2023).

Phenolic compounds can originate from natural processes which includes decay of organic matter, metabolism of certain plants. Human activities such as agro-industries and industrial processes also contribute to generation of phenolic compounds. Phenols are commonly used in the manufacture of plastics, polymers, construction, automotive, and appliance industries (Matike & Ngole-Jeme, 2024). Phenolic compounds are considered as priority contaminants due to their persistence in the environment. This is attributed to the stability of the structure of benzene ring (da Silva Aires *et al.*, 2024).

2.9.1 Natural sources of phenolic compounds

Phenols are released into to environment by natural sources such as plants which manufacture them as secondary metabolites to facilitate their existence. Plants may also release phenols during their decomposition. Some aquatic plants are known to

produce phenolic compounds as their metabolic by products. For example, marine algae are known to produce large molecules of phenolic structures (Ramos *et al.*, 2024).

Previous studies have reported that some organic compounds produce phenolic compounds such as plastics and plant materials when subjected to sunlight/UV. For example, exposure of hemicellulose to sunlight converts them to phenolic compounds (Nzimande *et al.*, 2024). This occurs as a benzene ring in cellulose decays under effect of sunlight. In addition, animals contain phenols in their metabolic excretions. Digestive system of mammals and humans converts tyrosine to phenolic compounds in their wastes Phenolic compounds are also present in some plant-based food such as fruits and vegetables. In addition, they may occur naturally in coal tar (Lv *et al.*, 2024; Rapiya *et al.*, 2025).

2.9.2 Industrial sources of phenolic compounds

Variety of industries such as pharmaceuticals, textiles and crude oil related industries produce phenolic compounds as final compounds or intermediates. Most of these compounds are then used in manufacture of farm chemicals, paints, resins and many others. These activities produce such as industrial effluents from manufacturing of pharmaceutical products, plastics, pesticides, crude oil processing, and refineries among others (Ramos *et al.*, 2024). Chemical spillage and leakages as well as marine accidents cause water, soil, and environmental pollution in general. In addition, coal gasification and wood distillation are major contributors of phenols in environment (Panigrahy *et al.*, 2022; Paun *et al.*, 2024).

2.9.3 Occurrence of phenolic compounds in water

The major source of phenolic compounds in water originates from plants as a result of biochemical reactions in presence of sunlight. Decay of dead plant materials such as leaves, roots and stem introduce these compounds to soil. Surface runoff washes them to the water sources (Rapiya *et al.*, 2025). Previous studies have reported presence of phenol, 2,4,6-trichlorophenol and 2,4-dinitrophenol in drinking (Santa-Cruz-Mérida *et al.*, 2025). This raises a lot of health concern. Most of these compounds originate from industrial and agricultural waste waters which flows into surface and ground water (Adjei *et al.*, 2021).

The presence of phenol compounds in water can also result from the chlorination process of water forming chlorinated phenolic compound. It has been observed that the use of water chlorine-based disinfectants results in the formation of different forms of phenols which are serious water pollutants (Parveen *et al.*, 2022; An *et al.*, 2023). Examples of phenolic compounds include 2-nitrophenol; 2,6-dichlorophenol; 3,4,5-trichlorophenol; 3-methylphenol; and 2,4-dimethylphenol to mention a few as shown by Figure 2.1.

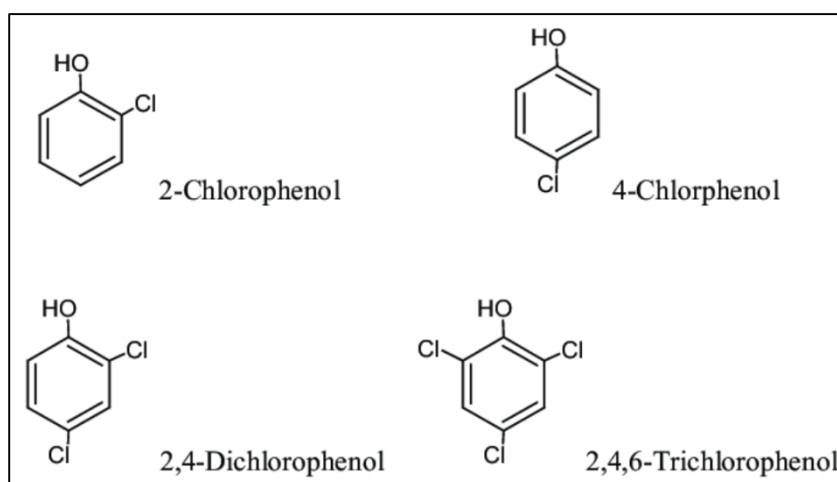


Figure 2.1: Some examples chlorinated phenolic compounds

During chlorination, at specific conditions, a substitution reaction occurs in the presence of halogen resulting in the formation of halophenol compounds as shown in sample reactions illustrated by Equation 2.3 and 2.4.



In addition, there is a chance of formation of aminated chloride compounds in water. These chlorination compounds react further with phenolic compounds in water resulting in a chloro-amination reaction. The halogenated phenolic compounds are a great environmental concern due to their potential toxicity (Santa-Cruz-Mérida *et al.*, 2025). Chloro-phenols are regarded as most toxic of all phenolic compounds. Bioaccumulation of phenolic compounds in water threatens the aquatic life and also agriculture and can result into contamination of all components of food chain (Adjei *et al.*, 2021).

Phenols are wide family of alkanols where hydroxyl groups are attached to benzene ring. Phenols which include hydro benzene, cresol, resorcinol, catechol, quinol, pyrogallol, hydroquinol, aminophenol among others. The structure of most common phenols as shown in the Figure 2.2.

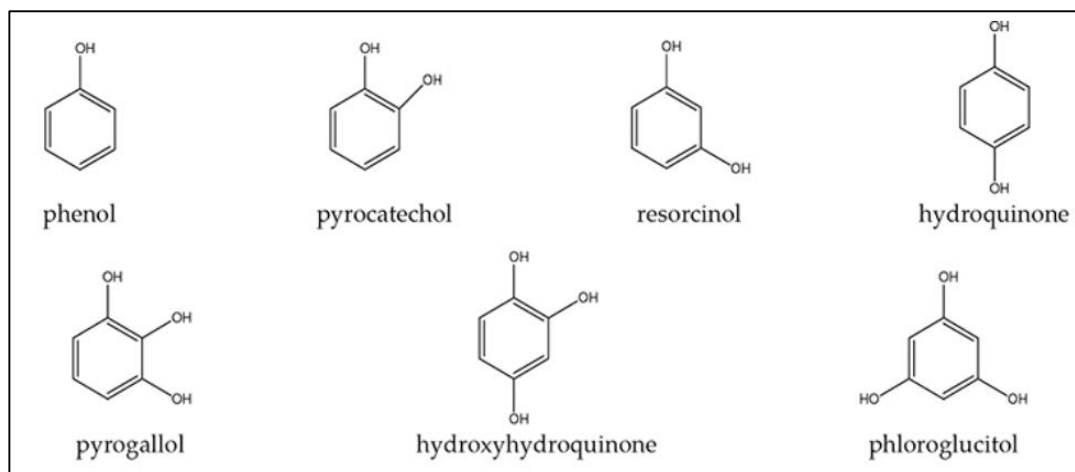


Figure 2.2: Some examples of phenols

Phenol and resorcinol which are also common phenols compounds. In this study, their adsorption from water was investigated.

2.10 Health effects of Phenolic compounds exposure

Human exposure to high levels of phenolic compounds is known to cause serious health challenges (Adjei *et al.*, 2021). According to WHO, the acceptable upper limit levels of phenolic compounds in water is 1 mg/L while most countries have set a maximum of 0.5 mg/L in drinking water (Li *et al.*, 2018; Ramos *et al.*, 2024).

On exposure to phenolic compounds may results in irritations to the eyes, skin, and eyes. Phenolic compounds are also known to cause muscle weakness and respiratory disorders such as irregular breathing, fluids accumulations in the lungs and impaired coordination when exposed by breathing (Ramos *et al.*, 2024). Consumption of water contaminated with phenolic groups has more serious negative effects on the health of the consumers. These include gastrointestinal damage, muscle tremors, walking abnormalities, and death. Most halogenated phenolic groups are highly mutagenic and are believed to be carcinogenic (Okoro *et al.*, 2022; Paun *et al.*, 2024). Phenolic compounds, especially 2,4,6-trichlorophenols and tetra-chlorophenols are ranked as in

3rd category of most severe carcinogens by The International Agency for Research on Cancer (IARC) (Adjei *et al.*, 2021; Parveen *et al.*, 2022).

On severe exposure to phenolic compound, the victim may initially exhibit symptoms such as nausea, headache, excessive sweating, ringing in the ear and dizziness. On severe exposure, the victim develops convulsions, sudden collapse, coma and even death (Ramos *et al.*, 2024). On long term exposure to phenolic compounds is associated with cardiovascular systems disorder such as high blood pressure which results in severe hypotension and shock, respiratory failure and death (Adjei *et al.*, 2021; Huang & Bu, 2022).

2.11 Remediation of residual aluminium ions

Dissolved metal ions in water can be removed by different techniques that are useful in the treatment of wastewater. These techniques include membrane filtration. This method suffers from membrane fouling due to accumulation of solid materials reducing membrane lifespan. Metal ions are removed at ultra-filtration or nano-filtration level (Azmi, 2025). Ion exchange, electrochemical degradation, photo-Fenton oxidation, reverse osmosis, and many others (Oladoye, 2022). Removal of residual metal ions in water can be achieved efficiently using maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticle adsorbents. This method is preferred due to its effectiveness, ease of operation, lack of secondary pollution, it is economical, among other advantages. Application of nanoparticles in adsorption is a preferred method due to their minute sizes and large surface area; they are effective in the remediation of contaminated water through adsorption. However, it has not been applied in adsorption of aluminium from water (Ethaib *et al.*, 2022; Shen *et al.*, 2019).

2.12 Remediation of phenolic compounds

The toxicological implications of exposure to phenolic compounds and their persistence in the environment bring the need to investigate efficient methods to eliminate these contaminants from drinking water (Paun *et al.*, 2024). Various methods have been used to remove phenolic compounds from water including adsorption, ozonation, biological methods and membrane separation (Adjei *et al.*, 2021). Effective removal of phenolic compounds from water, especially those contaminated with industrial effluents or urban waste waters helps in protecting aquatic life dependent on the water bodies from toxic effects of the phenols (Rapiya *et al.*, 2025). Phenols have

the most common water pollutants, originates mainly from industrial processes. These processes include papers and dye manufacturing, agricultural industries and municipal waste waters. Due to the oxidization, phenols are widely available in natural environment (Ramos *et al.*, 2024).

2.12.1 Removal of phenols compounds by adsorption

Adsorption is the method of choice in removal of phenolic compounds from contaminated water. Previous studies have investigated on different materials that can be used to adsorb these compounds from water. Activated carbon, Kaolin, bagasse ash among others. It was reported to have adsorption efficiencies of over 90% at optimum conditions. It was found that the rate of adsorption is affected by physical conditions such as pH and presence of other competing ions in the solution. Another study reported that activated carbon is an efficient and low-cost adsorbent of polyphenols from Olive-mill waste waters (Candan Eryilmaz & Ayten Genç, 2021; Mohd, 2022). Other advantages of using adsorption in remediation of phenolic compounds includes high efficiency, easy handling and flexibility in design (Lv *et al.*, 2024).

2.12.2 Removal of phenolic compounds by ozonation of phenolic groups

Ozonation is an oxidation processes which involves use of ozone (O₃) in removal of organic compounds such as phenols from water. During this process high oxidizing properties of ozone is utilized in decomposition of organic compounds into less harmful substances. A study by Otitoju *et al.* (2023) on removal of ethylene glycol from olive mill wastewaters reported that ozonation can achieve efficiency of up to over 80% in remediation of contaminated water. This process was found to be affected by change in conditions such as concentration of the phenolic compounds and gas-liquid contact time (Otitoju *et al.*, 2023).

2.12.3 Removal of phenolic compounds by biological methods

Biological methods involve degradation of phenolic compounds into non-toxic products by use of microorganisms. This process can be classified into two broad categories. That is, microbial and enzymatic methods. In microbial degradation some bacteria, fungi or yeast reacts with phenolic compounds under aerobic or anaerobic conditions. During biological methods, phenyl rings are broken down forming carbon(iv)oxide and water. Biological methods are preferred due to their low cost and adoptability to different physical conditions (Adjei *et al.*, 2021).

2.12.4 Removal of phenolic compounds by membrane separation

Membrane separation involves use of membranes of different pore sizes to remove phenolic compounds from water. Membrane separation can be classified into microfiltration, ultrafiltration and nanofiltration. The contaminants are separated based on size and concentration gradients. Therefore, the contaminants are first concentrated then later removed through additional subsequent treatments. The membrane separation techniques are influenced by pressure of the solution and its concentration gradient (Charcosset, 2021; Maroufi & Hajilary, 2023).

2.13 Nanoparticles

Nanotechnology is a science that involves study and applications of nano particles. These are objects of small sizes with diameters of between 100-2500 nm but are larger than an atom (Gupta, 2020; Singh & Jen, 2021). As result to their nano-scale, nanoparticles have a high surface area which allows very many different functional groups to be attached to them (Prerna & Ratan, 2021). Metal nanoparticles differ in both chemical and physical properties from those of bulk metals. For example, nanoparticles exhibit lower melting points, different specific optical properties and large surface area (Singh & Jen, 2021). Nanoparticles can be broadly metallic, ceramic, carbon-based, semiconductor, polymeric, and lipid-based nanoparticles (Wan *et al.*, 2023).

In modern times, there is a lot of advancement in nanotechnology. This has resulted in synthesis of more functionalized nanomaterials which have high adsorption efficiency. This is attributed to their particle sizes, surface charge and surface chemistry (Falke *et al.*, 2024).

2.13.1 Size of nanoparticles

Nanoparticles have very high specific surface area due to their small particle sizes. The boundary conditions of the crystalline particle changes when the particle size approaches the nano-scale. Sometimes the particle size may be smaller than wavelength of light. This makes nanoparticles to have physical properties different than the bulk materials (Altammar, 2023). The small particle size of the nanoparticles increases their adsorption capacity by increasing available adsorption sites for

interaction with metal ions. This facilitates both monolayer and multilayer adsorption (Karnwal & Malik, 2024; Olawade *et al.*, 2024).

2.13.2 Surface charge of nanoparticles

Adsorption of metal ions is significantly dependent on surface charge of the nanoparticles. The electro-kinetic characteristics of nanoparticles such as zeta potential greatly influence their stability and adsorption of metal ions. Previous studies have reported that surface modification alters the surface charge properties improving their binding tendency to the metal ions. Varying surface charge tends to either promote or hinder adsorption capacity of adsorbent based on the ionic characteristics of the surrounding environment (Bilardo *et al.*, 2022; Olawade *et al.*, 2024).

2.13.3 Surface chemistry of nanoparticles

Surface characteristics of nanoparticles is a major factor in determination of adsorption efficiency when in contact with metal ions. Additionally, specific functional groups in surface modification of nanoparticles, have been studied by different researchers in enhancing adsorption capacity of the nanoparticles. Examples of such groups include, amino, nitro, thiol and carboxyl among other groups. This improves their efficiency and selectivity in adsorption of particular metal ions (Bilardo *et al.*, 2022; Olawade *et al.*, 2024). This surface functionalization enhances the bonding strength between metal ions of interest and the nanoparticles by formation of stable complexes (Manyangadze *et al.*, 2020). For example, carbon nanotubes modified with carboxyl groups exhibited improved adsorption selectivity for lead, mercury and cadmium ions. This is attributed to formation of strong metal-carboxylate binding (Olawade *et al.*, 2024; Raji *et al.*, 2023).

2.14 Applications of nanoparticles in water remediation

Nanotechnology has grown tremendously in recent times and has been applied in numerous small-scale and industrial processes. Nanoparticles have a very large surface area due to their small size. Due to this advantage, they have wide applications in modern times. Nanoparticles were first used around the 16th century as a medicine in the form of colloidal gold to treat various diseases such as dysentery, tumors, and epilepsy (Kowalska *et al.*, 2024). Nanometals are commonly applied in the adsorption of metals and other non-metal ions such as phenolic compounds. These substances are

significant environmental pollutants due to their bio-accumulation properties harming human health and other animals. Their toxicity is magnified by being non-biodegradable and not metabolized or decomposed by human bodies (Adjei *et al.*, 2021). The application of metal nanoparticles in the adsorption of water pollutants is preferred due to their low cost, friendly to the environment, and high catalytic activities (Raji *et al.*, 2023).

Adsorption can be classified as physisorption due formation of weak van der Waals forces between adsorbate and adsorbents or chemisorption as a result of covalent or ionic bonds (Murage *et al.*, 2022). Aluminium ions exposure to humans can be attributed to the consumption of contaminated water among other sources. Therefore, there is a need to find an efficient and practical method in water remediation by removal of the undesirable metal ions from water. Nanoparticles have large surface area to mass ratio and high adsorption capacity. Therefore, are conveniently used in the adsorption of metal ions and other contaminants from water or soil.

The adsorption capacity of nanoparticles are greatly influenced by the size of the particles, porosity, physical structure, and chemical composition of the nanoparticles (Olawade *et al.*, 2024). In the removal of metal ions from water, the most common nanoparticles adsorbents include metals in zero-valent state magnetic nanoparticles, iron-based nanoparticles, chitosan nanofibers, Fe₃O₄, MnO₂, CNPs, AC, Graphene, TiO₂, zero-valent iron nanoparticles, and many others (Makhanya, 2020; Manyangadze *et al.*, 2020; Raji *et al.*, 2023).

2.15 Maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles adsorbent

Maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles has become one of the most sort after nano adsorbent in recent studies. this is attributed to it special properties such non- toxic, superior hysteresis properties, thermal and chemical stabilities among others (Lemine *et al.*, 2021). Iron oxides are some of the most common nanoparticles used metallic-based nanoparticle adsorbents. They may be used in their unsupported (naked) form or supported by other materials such as polymers, MnO₂, ZnO, TiO₂, or polymer composites (Ali & Khan, 2020). Iron-based nanoparticles can be in the form of superparamagnetic iron oxides like, magnetite (Fe₃O₄), maghemite($\gamma\text{Fe}_2\text{O}_3$) and hematite($\alpha\text{Fe}_2\text{O}_3$) (Iqbal *et al.*, 2025). Maghemite is a ferromagnetic iron oxide which predominantly has oxidation state of iron (iii). These maghemite nanoparticles have

high magnetic properties with an average particle size of 9 nm. They high adsorption capacity therefore make efficient adsorbents in water remediation by removal of metallic ions and other water contaminants (Busti *et al.*, 2021; S. Kumar *et al.*, 2021).

Previous studies on water remediation reported that that Maghemite is an efficient adsorbent of arsenic metal ions from water. In addition, maghemite was found to be very effective adsorbent in removal of Cu^{+2} and Mn^{+2} from water. It was found that adsorption efficiency was affected by change in contact time, change in the initial concentration of adsorbent, change in pH and presence of other ions such as SO_4^{-2} ions (Kumar *et al.*, 2021; Mabudi *et al.*, 2025). In a study carried out by (Maneechakr and Mongkollertlop (2020) maghemite was used in water remediation by adsorption of Cr^{+3} , Cd^{+2} and Hg^{+2} ions. It was reported that percentage adsorption of the metal ions was influenced by change in pH, temperature, concentration of adsorbate and adsorbent dosage (Maneechakr & Mongkollertlop, 2020).

In another study carried out by Arul *et al.* (2023), they reported that magnetic maghemite is an efficient adsorbent of Pb^{+2} and Cd^{+2} ions from water. Adsorption was carried out using functionalized maghemite nanoparticles prepared by alkaline co-precipitation. The surface of adsorbent was functionalized using fatty and amino acids, carbon nanotubes and silica. The study reported that the type of modification done on the adsorbent had a large effect on the overall adsorption capacity. In addition, the rate of adsorption increased with increase in adsorbent dosage (Arul *et al.*, 2023).

2.16 Other properties of maghemite ($\gamma\text{Fe}_2\text{O}_3$)

Maghemite nanoparticles exhibits properties similar to other iron oxide nanoparticles such as hematite and magnetite. The formula of maghemite ($\gamma\text{Fe}_2\text{O}_3$) differ from that of magnetite (Fe_3O_4) which can also be expressed as $\text{FeO} \cdot \text{Fe}_2\text{O}_3$. In addition, the structure of maghemite is octahedral structure with iron vacancies while magnetite does not exhibit similar vacancies (Coduri *et al.*, 2020). However, variations in properties may be observed. These variations include;

2.16.1 Magnetic properties

Maghemite nanoparticles are ferrimagnetic with magnetic moments of $2.5 \mu_B/\text{unit}$ formula (Shokrollahi, 2017). Maghemite has phase transition temperature of between 500 to 900 Kelvin. This make maghemite ($\gamma\text{Fe}_2\text{O}_3$) and magnetite (Fe_3O_4)

nanoparticles to exhibit close magnetic properties due to similar structure and chemical composition (Andronenko *et al.*, 2023). Iron ions in maghemite are arranged in a spinel structure giving it strong magnetic characteristics when exposed to external magnetic field (Poh Yan *et al.*, 2022). These magnetic properties of maghemite nanoparticles significantly enhances their characteristics as adsorbents. This property facilitates efficient recovery of nanoparticles after adsorption. This property is very important in remediation of water contaminated with hazardous metals such as lead, arsenic, chromium and cadmium (Arul *et al.*, 2023; Olawade *et al.*, 2024).

2.16.2 Small particle size

Nanoparticles are known for their very small particle sizes. Adsorption capacity is greatly affected by the physicochemical properties of the nanoparticles. This is because particle sizes and surface properties determine the quantity, type and strength of bonds formed between the adsorbate and nanoparticles. In general, smaller particles have higher mobility and improved surface reactivity enhancing adsorption capacity. Maghemite has particle sizes of diameter of about 9 nm (Iqbal *et al.*, 2025; Youssif *et al.*, 2024).

2.16.3 Large surface area

Maghemite nanoparticles have very high surface area which provides large quantity of adsorption sites. The adsorption properties of the nanoparticles can be improved by modification involving attaching other functional groups. Previous studies have taken advantage of these properties of nanoparticles in remediation of contaminated water by removal of metallic and other non-metals such as phenolic groups from water (Bilardo *et al.*, 2022; Xiao *et al.*, 2023; Youssif *et al.*, 2024).

2.17 Adsorption mechanism of maghemite ($\gamma\text{Fe}_2\text{O}_3$)

Adsorption involves interaction between nanoparticles and the adsorbates. The success of adsorption is influenced by various physicochemical factors such as particle size, surface area, surface charge and morphology of the nanoparticles (Fawzy *et al.*, 2024; Manyangadze *et al.*, 2020). There is formation of strong electrostatic bond between maghemite nanoparticles and metal ions. Previous studies have reported that decrease in pH results in reduced adsorption efficiency of metal ions due to competition for adsorption sites with H^+ ions in the solution (Iqbal *et al.*, 2021).

In a study on adsorption of Diclofenac using maghemite, it was reported that adsorption occurred in two stages. In the fast initial step, there was formation of a bond between nanoparticles and diclofenac molecules. The second step occurred slowly and it involved diffusion of the adsorbate molecules into pores of the maghemite (ALothman *et al.*, 2020; Şahin *et al.*, 2022). In another study involving adsorption of Eu^{+3} using synthetic maghemite, it was reported that adsorption mechanism primarily involved formation of electrostatic bonds and also surface complexation (Hassan & Aly, 2021; Heiba *et al.*, 2022). Maghemite nanoparticles has been used in adsorption arsenite from water. It is preferred to other adsorbents due to its high adsorption capacity, large surface area, non-toxicity and chemical stability (Ranjan *et al.*, 2022; Boruah *et al.*, 2023). In this study, efficiency of adsorption residual aluminium and phenolic compounds from water using maghemite nanoparticles is studied.

2.18 Synthesis of maghemite ($\gamma\text{Fe}_2\text{O}_3$)

Maghemite ($\gamma\text{Fe}_2\text{O}_3$) can be synthesized conveniently using various methods. These techniques include, co-precipitation, sol-gel, hydrothermal, green synthesis among others. Each of these methods have their differences, advantages and disadvantages as discussed here below.

2.18.1 Co-precipitation

Most common method is chemical precipitation. This method is preferred due to it control of nanoparticle sizes and their distribution. In addition, this method is convenient due to it simple procedure, low cost, high purity and high yield giving large amount of nanoparticles with low oxidation rate. These properties are important in maximizing adsorption capacity of the nanoparticles (Gutierrez *et al.*, 2024; R. Kumar *et al.*, 2021). Precipitation occurs when the solubility product of a compound of interest is exceeded. In this method, a stoichiometric mixture of Fe^{+2} and Fe^{+3} salt is precipitated using an alkali. Most commonly used alkalis include sodium hydroxide, potassium hydroxide or ammonium hydroxide. The preferred salts include sulphates, nitrates, and chlorides. In this method, the particle size and surface characteristics are influenced by variables such as pH and reaction temperature (Iqbal *et al.*, 2021).

2.18.2 Sol-gel

This method involves a hydrolysis reaction of metal alkanoxides using an acid or base. The synthesized nanoparticles are then dried and prepared into sol-gel (Bokov *et al.*,

2021). For example, homogeneous solution of metal complexes are hydrolyzed before been converted into organo-polymers. These are later dried to produce xerogels and aerogels. This method has advantage of having simple synthetic route, produces highly porous nanoparticles and under normal processing temperatures. In addition, the nanoparticles formed have consistent particles sizes resulting in uniformity in the properties (Benamara *et al.*, 2023; Lemine *et al.*, 2021).

2.18.3 Microemulsion

Microemulsions are thermodynamically stable isotropic colloidal dispersions. They have polar, non-polar and surfactants domains (Mitra, 2023). In this method, nanoparticles are prepared by reaction of iron salt precursors solution with surfactants or co-surfactants. Then the iron oxide nanoparticles are precipitated using ammonia to the microemulsion. Sometimes water-in-oil microemulsions are used (Du & Hou, 2026).

2.18.4 Hydrothermal synthesis

In this method Fe^{+3} and Fe^{+2} salts solutions are formed by dissolving in de-ionized water separately. These solutions are then mixed under constant stirring. Precipitation is done by addition of sodium hydroxide. The precipitates are dried in an oven at about $150\text{ }^{\circ}\text{C}$. Maghemite nanoparticles formed are then washed in ethanol (Nnadozie & Ajibade, 2022; Silva *et al.*, 2023).

2.18.5 Green synthesis

The green method is where nanoparticles are prepared using plant materials. This method is preferred because it simple and low cost. In addition, it is environmentally friendly since it does not use hazardous chemicals (Aida *et al.*, 2023; Siddiqi *et al.*, 2025). In green synthesis of various plant extracts and biological substances have been used by different researchers. Plant extracts contain antioxidants that reduce metal oxides resulting in the precipitation of nanoscale particles. These bio-chemicals include polyphenols, phenolic acids, and flavonoids among other compounds (Kumar *et al.*, 2021; Piro *et al.*, 2022). In a study carried out on preparation of magnetic iron oxide nanoparticles by the green method. He reported that seaweed (*Sargassum muticum*) plants extract contained sulphated polysaccharides which effectively reduced iron (iii) chloride-forming magnetite (Swathi *et al.*, 2024).

Wirwis and Sadowski (2023), in preparation of gold and silver nanoparticles using the green method found that green tea extract contained many anti-oxidants. These reducing agents included gallic acid and epigallocatechin gallate compounds (Wirwis & Sadowski, 2023). In addition, Rostamizadeh *et al.*, (2020) used Cornelian cherry fruit extract in the synthesis of iron oxide nanoparticles. In a study by Hu *et al.*, (2025), Iron (iii) nitrate was reduced using extracts prepared from green tea to synthesize polyphenol-iron nanoparticles. Bhuiyan *et al.*, (2020) synthesized $\alpha\text{Fe}_2\text{O}_3$ from hydrated iron (iii) chloride which was reduced by papaya leaf extract. This study was done at conditions of room temperature and an alkaline environment of pH value 11.

The bio-compounds are effective reducing agents used in synthesis of stable nanoparticles. The plant extracts contain different functional groups which includes carboxyl, hydroxyl and aldehydes in the plant extract reduces, stabilizes and acts as the capping agents on the iron salts (Bhuiyan *et al.*, 2020; Ekwumemgbo *et al.*, 2023). Chemical and physical methods such as co-precipitation, hydrothermal and sol-gel uses toxic chemicals and reagents. Therefore, green synthesis is the method of choice in preparation of nanoparticles due to its low toxicity, low-cost efficiency and its friendly to the environment (Miri *et al.*, 2023).

2.19 Characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using XRD

X-ray diffraction (XRD) is an efficient and effective analytical technique used to provide accurate information on the composition, crystal structure, and particle size of nanoparticles (Chhantyal, 2022). A German physicist Wilhelm Conrad Röntgen discovered X-rays radiations in 1895.

2.19.1 Working principle of XRD

X-Ray is a form of electromagnetic radiation in a spectrum. It is expressed in terms of parameters such as frequency, energy and wavelength. It has a wavelength range of 1nm^{-1} pm which is smaller than visible light. The rays are generated by a cathode ray tube before been passed through screens and filters to form a monochromatic X-ray beam which is then focused onto the analyte. A diffraction is created when the X-ray beam interacts with the crystals of the analyte. Diffraction is where crystals are scattered at specific angles by crystals due to the atom arrangement in a crystal (Ahmad *et al.*, 2021; Kumar *et al.*, 2021; Mongkolsuttirat & Buajarn, 2021).

2.19.2 Components of XRD

X-Rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target (tungsten) by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. Filtering, by foils or crystal monochromators, is required to produce monochromatic X-rays needed for diffraction. These X-rays are collimated and directed onto the sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded. When the geometry of the incident X-rays impinging the sample satisfies the Bragg Equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor.

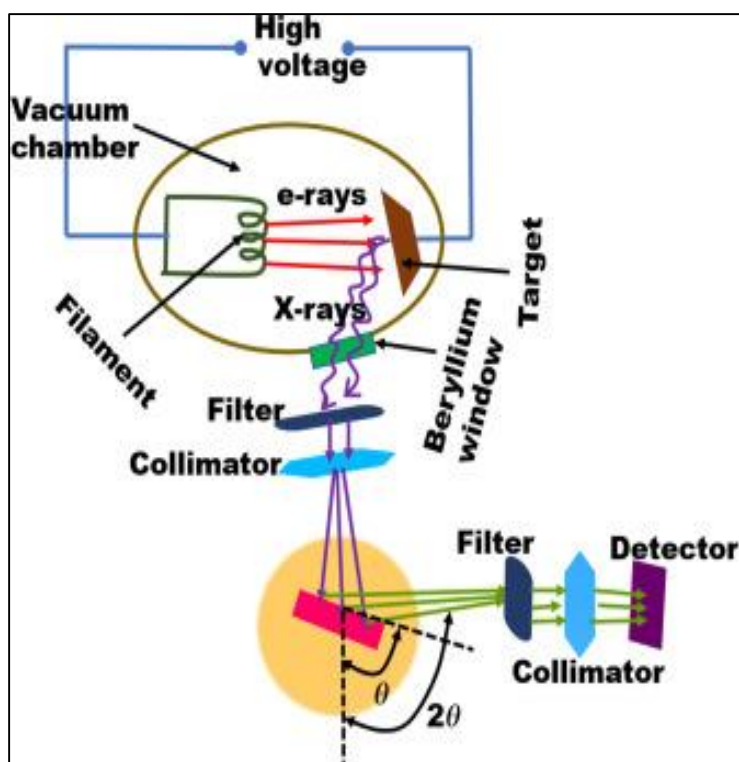


Figure 2.3: Cross-section diagram of XRD

XRD is one of the most efficient analytical technique in determination of crystallite size of nanoparticles. The broadening of diffraction peaks are obtained from the constructive interference of the scattered X-ray at specific angle from each set of

lattice planes of the nanoparticle sample. Therefore, the diffraction peak broadening can be analyzed to estimate the average crystallite size of the nanoparticles (Mongkolsuttirat & Buajarern, 2021; Chhantyal, 2022; Surdu & György, 2023).

2.20 Characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using UV-Vis

UV-Vis is a spectral-analytical technique based on the measurement of characteristics of UV rays after interacting with the analyte.

2.20.1 Working principle of UV-Vis

It involves the determination of the number of discrete wavelengths of UV or Visible light transmitted through or absorbed by the analyte in comparison to a blank sample or reference sample. Valuable information about the analyte's composition and concentration is obtained based on the characteristics of wavelengths detected after interaction with the sample. In this technique, the absorption of electromagnetic radiation occurs at wavelength range of 190 to 800 nm (Wu *et al.*, 2021; Surkova *et al.*, 2022).

UV-Vis spectroscopy technique is commonly applied in the quantitative analysis of different types of organic, inorganic, and biological compounds. It is also applied in many areas of scientific fields such as, determination nuclei acid, biological culturing, drug characterization, quality control of industrial products and in scientific research (Galieva *et al.*, 2023; Ferreira *et al.*, 2025).

2.20.2 Components of UV-Vis

UV-Vis consists of mainly, light source, monochromator absorption filters, sample holder data processor and computer as shown in Figure 2.4.

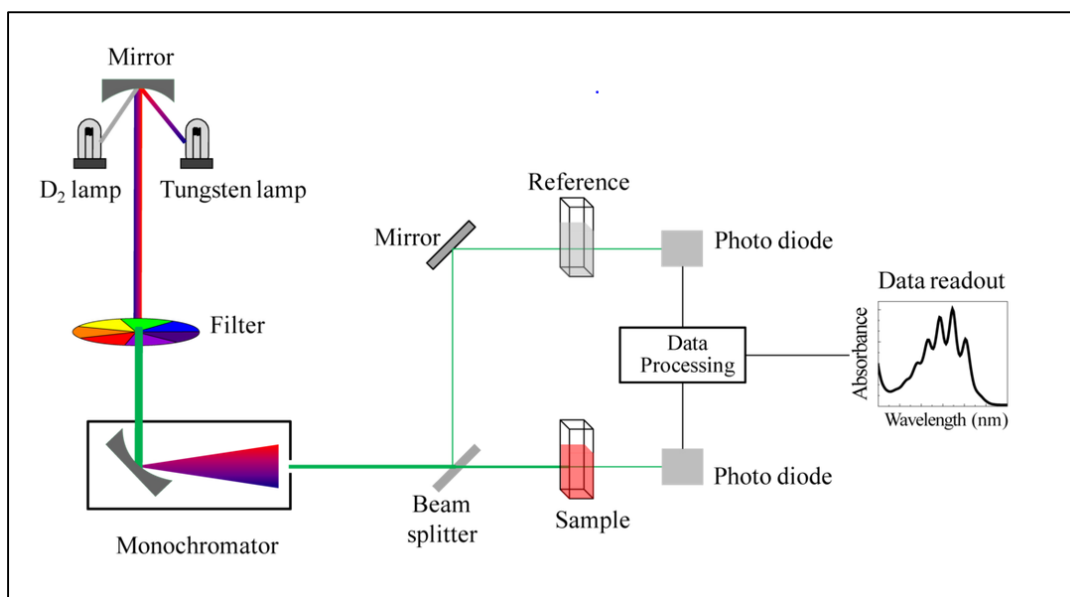


Figure 2.4: Cross-section diagram of UV-Vis

Single xenon lamp is commonly used as light source. It should be a high intensity light source for both UV and visible light range. Limitations of xenon lamps includes, are expensive and are less stable. Some spectrometers make use of two lamps Deuterium lamp for UV light and Tungsten or Halogen lamps for visible light. switching between the two lamps occurs at 300 to 350 nm for smooth scanning.

The wavelength selector picks the light wavelength suited for the analysis of the specific analyte. The methods of wavelength selection include; Monochromators which separates light into narrow bands of wavelength using diffraction gratings which are rotated to choose a particular desired wave length. Absorption filters are commonly made of colored glass or plastic designed to absorb particular wavelengths of light. Interference filters, these are layers of dichroic filters are used to eliminate undesirable wavelengths by destructive interference. Other wavelength selectors include Cutoff filters and Bandpass filters. Monochromators and filters are usually used together for more precise measurements and to improve the signal-to-noise ratio.

During sample analysis, light from the wave selector is passed through the sample analyte as well as the reference (Blank sample). The reference signal is used to determine the absorbance of the analyte. Signal detector is used to convert the light into a readable electronic signal. Generally, detectors are based on photoelectric coatings or semiconductors. A photoelectric coating ejects negatively charged

electrons when exposed to light. A photomultiplier tube (PMT) one of the more common detectors used in UV-Vis spectroscopy. Semiconductors are exposed to light, an electric current proportional to the light intensity can pass through. After the electric current is generated from whichever detector was used, the signal is then recognized and output to a computer or screen (Wu *et al.*, 2021; Shi *et al.*, 2022; Surkova *et al.*, 2022; Vogt *et al.*, 2023).

2.21 Characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using FTIR

FT-IR stands for Fourier Transform InfraRed, the preferred method of infrared spectroscopy.

2.21.1 Working principle of FTIR

In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample.

Fourier Transform Infrared Spectroscopy (FTIR) is an important technique that provides a fast and robust way to identify the presence of certain functional groups in an organic molecule. Functional groups have vibration frequencies that are characteristic of the target functional group. These vibration frequencies fall within the infrared (IR) frequency range. As such, passing an IR signal through the organic compound causes the functional groups to vibrate at specific frequencies. In other words, an infrared signal that passes through an organic compound will be absorbed at these characteristic frequencies, which can be transformed into a unique spectrum (Eid, 2022; Pasieczna-Patkowska *et al.*, 2025; Wadhwa *et al.*, 2022).

2.21.2 Components of FTIR

FTIR consist mainly of IR radiation source, interferometer, sample holder, detector and a computer readout as illustrated by Figure 2.5.

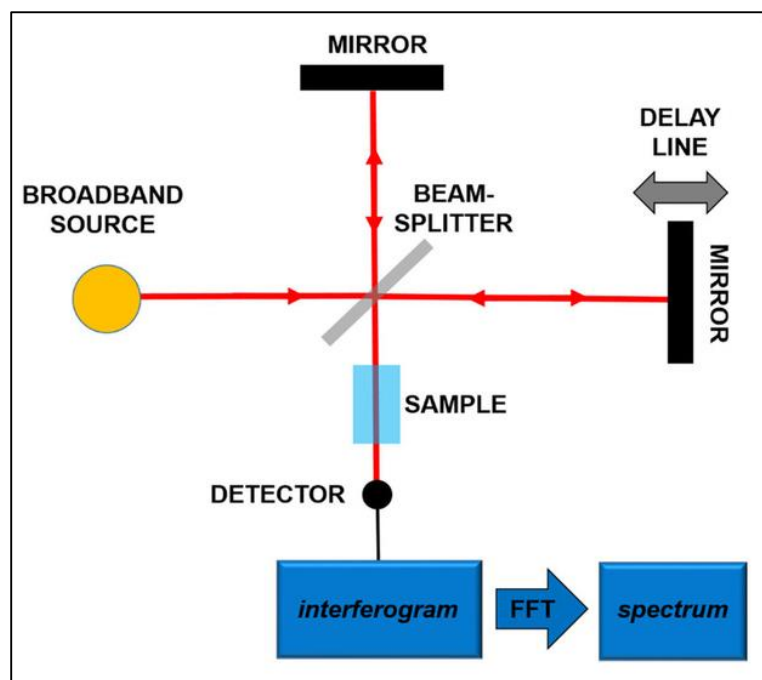


Figure 2.5: Cross-section diagram of FTIR

Infrared energy is emitted from a glowing black-body source. This beam passes through an aperture which controls the amount of energy presented to the sample and, ultimately, to the detector. Examples of IR sources includes; Tungsten filament lamp, Nernst glower, Globar, Mercury arc and Nichrome coil (Singla *et al.*, 2022).

The beam enters the interferometer where the “spectral encoding” takes place. The resulting interferogram signal then exits the interferometer. Interferometer is an optical device which produces a unique type of signal which has all of the infrared frequencies “encoded” into it. The signal can be measured very quickly, usually on the order of about one second. Most interferometers employ a beamsplitter which takes the incoming infrared beam and divides it into two optical beams. One beam reflects off of a flat mirror which is fixed in place. The other beam reflects off of a flat mirror which is on a mechanism which allows this mirror to move a very short distance (typically a few millimeters) away from the beamsplitter. The two beams reflect off of their respective mirrors and are recombined when they meet back at the beamsplitter. Because the path that one beam travels is a fixed length and the other is constantly changing as its mirror moves, the signal which exits the interferometer is the result of these two beams “interfering” with each other. The beam enters the sample compartment where it is transmitted through or reflected off of the surface of the

sample, depending on the type of analysis being accomplished. This is where specific frequencies of energy, which are uniquely characteristic of the sample, are absorbed (Singla *et al.*, 2022; Pasieczna-Patkowska *et al.*, 2025).

The beam finally passes to the detector for final measurement. The detectors used are designed to measure the special interferogram signal. Types of detectors include; Thermal detectors, Photo-conducting detectors (Singla *et al.*, 2022). The measured signal is digitized and sent to the computer where the Fourier transformation takes place. The final infrared spectrum is then presented to the user for interpretation and any further manipulation (Wadhwa *et al.*, 2022).

In dispersive IR spectroscopy monochromatic light sequentially irradiates a sample spanning an infrared wavelength range, FTIR spectroscopy irradiates a sample with many frequencies of IR light at once, utilizing an interferometer and post-processing of the transmitted light. A beam source of various IR wavelength light is sent through a beam splitter, where half reaches a fixed mirror and half a mirror that moves with a constant velocity. These two split beams are then reflected and recombined (now with a path difference between the beams) to construct an interference pattern reflecting the constructive and destructive interference of the recombination. After, this interference pattern (or interferogram) is sent to the sample, and the transmitted portion of the interferogram is sent to a detector.

After comparison with a reference sample beam spectrum in the detector, a Fourier transform is performed to obtain the full spectrum as a function of wavenumber. The IR spectrum can be presented either as absorbance or transmission. The transmittance is typically presented as downward peaks in an IR spectrum, while absorption is an upward peak (Eid, 2022; Singla *et al.*, 2022; Surkova *et al.*, 2022).

2.22 Determination of concentration of residual aluminium ions by AAS.

Atomic absorption spectroscopy (AAS) is an efficient analytical technique that is widely used in detection and determination of concentration of metal ions in aqueous solutions. This technique is widely used for precise identification and quantification of metallic analytes in environmental monitoring, clinical research and food safety control. AAS analysis involves absorption light energy of specific wavelength to the by free atoms of given element. This technique is very sensitive and can detect very

low metal ion concentrations down to 0.0001 ppm using flame procedure (Abrham & Gholap, 2021; Farooq & Jalees, 2020).

2.22.1 Working principle of AAS

A liquid analyte is introduced at high pressure through the nebulizer. It is aspirated and mixed with fuel and oxidant before being ignited. This is done using graphite furnace or acetylene/ air flame where analyte is atomized. A beam of UV light of particular wavelength. Hollow cathode lamp is commonly used as the source of radiation. As atoms absorb the radiation, electrons in the atoms are elevated to excited state. The amount of light absorbed is proportional to the concentration of the analyte. The detector measures the variance in the intensity of the illuminated light. A computer system converts the change in intensity into absorbance. The absorbance is directly related to the concentration of the element in the sample (El-Dib *et al.*, 2020; Idris *et al.*, 2021; Nejati *et al.*, 2022).

2.22.2 Components of AAS

Hollow cathode lamp is the most commonly used as source of radiation. However, electrode discharge lamps are also used. The radiation produced is used to is at specific wavelength corresponding to the electronic transition of the target element. Atomizer is also referred to as nebulizer and it is important in converting the analyte sample into atomic form. This involves breaking chemical bonds within compounds forming free atoms which can conveniently absorb illuminated radiation. In flame atomic absorption spectroscopy, flame is used to vaporize the sample. While in graphite furnace atomization, a furnace is used to achieve high temperatures. This is important in improving sensitivity of the analysis (Abrham & Gholap, 2021; Gandhi *et al.*, 2022).

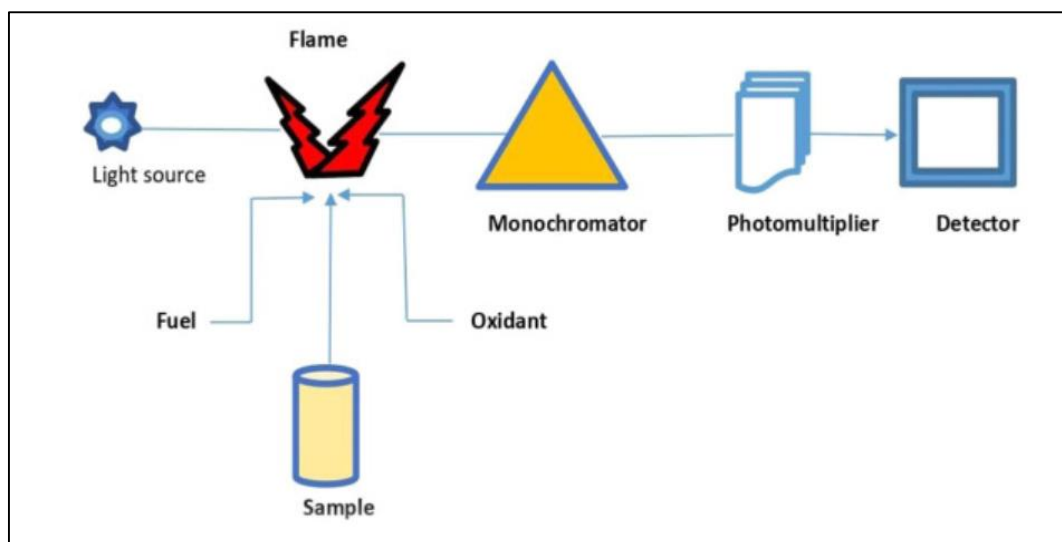


Figure 2.6: Cross-section diagram of AAS

Monochromator is used to isolate the specific wavelength of light that corresponds to the metal analyte of interest. Prisms and grating monochromators which isolates the radiated light to allow only the relevant wavelength to pass to the detector. Detector measures the variance in the intensity of the illuminated light after passing through the sample. Commonly detectors in flame atomic absorption spectrometers includes photomultiplier tubes and solid-state detectors. The detectors convert the light signal into an electrical signal that is directly proportional to concentration of the analyte. The computer system processes the electrical signal from the detector and displays the results in a quantifiable data. The results include, flame intensity, absorbance and concentration values which are crucial in interpretation of the results (Farooq & Jalees, 2020; Idris *et al.*, 2021; Gandhi *et al.*, 2022; Nejati *et al.*, 2022).

2.23 Adsorption isotherms models

The adhesion of the particles of adsorbate on the surface process is called adsorption. This causes adsorbate particles to be deposited on the surface of the adsorbent due to the removal of the adsorbate from the liquid phase. Adsorption isotherms illustrate the adsorption process of a surface with respect to a specified temperature. It describes the amount of adsorbate bound onto the adsorbent surface as a function of the concentration of adsorbate in the solution (Ehiomogue *et al.*, 2021; Kalam *et al.*, 2021).

The adsorption models exist that are based on the distribution of the adsorbate on the adsorbent. The easiest-to-use method to determine the most suitable adsorption model is linear regression analysis. Various adsorption models like Langmuir, Freundlich, Temkin, Dubinin-Rudashkevich, linear, Brunauer Emmett and Teller (BET) adsorption model, etc., are used (Abin-Bazaine *et al.*, 2022; Xia *et al.*, 2022). The adsorption isotherm models are mathematical equations that give the relationship between the amount of the adsorbate (q_e) on the surface of the adsorbent and the concentration of the adsorbate in solution (C_e) at a fixed temperature. The isotherm models used to fit the adsorption of phenol and resorcinol on Maghemite in this study are four isotherm models (Wang & Guo, 2020).

2.23.1 Langmuir adsorption isotherm

One of the most widely used models to describe adsorption is the Langmuir isotherm model, which was proposed by Irving Langmuir in 1918. The assumption is that the surface is homogeneous, the number of the adsorption sites is small, and that only one molecule can be accommodated into each site (Monolayer adsorption). It also assumes that there is no interaction between the molecules that are adsorbed, and that the energy of adsorption is the same all over the surface (Xia *et al.*, 2022; Yuan *et al.*, 2022).

The main assumptions of the Langmuir model are: Only adsorption in the first, or monolayer, layer occurs on the surface, there are no interactions between adsorbed molecules (no lateral interactions), the surface is homogeneous, that is, each molecule encounters the same adsorption energy. Langmuir equations can be linear or non-linear equations. The Langmuir linear isotherm equation (used in this study) is shown in Equation 2.5.

$$C_e/q_e = 1/(q_{\max} \cdot K_L) + C_e/q_{\max} \quad (\text{Equation 2.5})$$

The Langmuir non-linear isotherm equation is shown in Equation 2.6.

$$q_e = (q_{\max} \cdot K_L \cdot C_e) / (1 + K_L \cdot C_e) \quad (\text{Equation 2.6})$$

From the above equation (equations 2.5 and 2.6), the symbol " q_e " denotes the amount of adsorbate adsorbent that can be adsorbed onto the unit mass of adsorbent at

equilibrium (mg/g). “Ce” is the equilibrium concentration of the adsorbate in solution (mg/L). The symbol “q_{max}” denotes the maximum adsorption capacity at a single layer (mg/g) of the adsorbent, and the symbol “KL” denotes the langmuir adsorption constant, which is related to the energy of adsorption and binding affinity (L/mg). The plot of Ce/q_e as a function of Ce is linear, where q_{max} (slope = 1/q_{max}) and KL (intercept = 1/(q_{max} · KL)) are obtained.

The Langmuir isotherm can also be expressed in terms of a dimensionless separation factor, RL, which indicates the favourability of adsorption as expressed by Equation 2.7 for the Langmuir separation factor.

$$RL = 1 / (1 + KL \cdot C_0) \quad (\text{Equation 2.7})$$

Where C₀ is the initial adsorbate concentration. The Value of RL indicates the type of adsorption, as shown in Table 2.1 below (Abin-Bazaine *et al.*, 2022; Amola *et al.*, 2022; Xia *et al.*, 2022).

Table 2.1: Value of RL versus type of adsorption

RL Value	Adsorption Type
RL > 1	Unfavourable
RL = 1	Linear
0 < RL < 1	Favourable
RL = 0	Irreversible

In the Langmuir isotherm, parameters are calculated by plotting a graph of Ce/q_e (y-axis) vs Ce (x-axis). The slope gives 1/q_{max}, and the y-intercept gives 1/(q_{max} · KL) (Amola *et al.*, 2022).

2.23.2 Freundlich adsorption isotherm

Freundlich isotherm is an empirical equation that represents adsorption on a heterogeneous surface, in which the non-uniform distribution of heat of adsorption and binding energy (Yuan *et al.*, 2022). The adsorption does not assume the saturation

of the adsorbent, and it permits the formation of multilayer adsorption. It has been widely used in aqueous or non-aqueous systems (Xia *et al.*, 2022).

The Freundlich model is used to represent adsorption on energetically heterogeneous surfaces. Does not consider the saturation limit because it can be a multilayer adsorption. Empirical - parameters obtained experimentally. It is often observed in systems in which the surface chemistry of the adsorbent is complex. Freundlich adsorption isotherm model can be linear (used in this study) or non-linear.

The Freundlich linear adsorption isotherm model is given by Equation 2.8.

$$\log(q_e) = \log(KF) + (1/n) \cdot \log(C_e) \quad (\text{Equation 2.8})$$

Freundlich non-linear adsorption isotherm model is given by Equation 2.9.

$$q_e = KF \cdot C_e^{(1/n)} \quad (\text{Equation 2.9})$$

The Freundlich adsorption isotherm equations are written as: in the above equation, the symbol " q_e " indicates the quantity of adsorption (mg/g) at equilibrium. " C_e " is the equilibrium concentration in solution (mg/L). KF is the Freundlich constant related to adsorption capacity (mg/g), and " $1/n$ " is the heterogeneity factor, which shows the adsorption intensity (dimensionless). " n " represents the adsorption intensity constant.

The intercept is $\log KF$, and the slope is $1/n$. A plot of $\log(q_e)$ vs. $\log(C_e)$ results in a straight line from which the constants KF and n can be determined. Values of $1/n$ during adsorption can be interpreted as shown in Table 2.2 below (Vigdorowitsch *et al.*, 2021; Xia *et al.*, 2022; Yuan *et al.*, 2022).

Table 2.2: Interpretation of the 1/n Value

1/n Value	Implication	Surface Type
$1/n < 1$	Favourable adsorption	Heterogeneous surface
$1/n = 1$	Linear/ideal adsorption	Homogeneous surface
$1/n > 1$	Unfavourable/cooperative	Complex interactions

In the Freundlich adsorption model, parameters are determined by plotting a graph of $\log(q_e)$ (y-axis) vs $\log(C_e)$ (x-axis). The slope gives $1/n$, and the y-intercept gives $\log(KF)$ (Yuan *et al.*, 2022).

2.23.3 Temkin adsorption isotherm

The Temkin isotherm was developed by Temkin and Pyzhev (1940), and it is assumed that the heat of adsorption of all the molecules in the layer is linearly reduced with coverage due to the interaction between the adsorbate and the adsorbent (Olasehinde *et al.*, 2020). The Temkin isotherm differs from the Langmuir and the Freundlich models by taking into account the interaction between the adsorbate and the adsorbent, which is indirect. It is based on the assumption that the binding energies are equally distributed up to the maximum binding energy. It can be used to indicate the heat of adsorption and interactions between the adsorbate and surface (Xia *et al.*, 2022). To calculate the Temkin linear adsorption isotherm, a linear Form equation (used in this study), as shown by Equation 2.10, is used (Mudhoo, 2025).

$$q_e = B \cdot \ln(AT) + B \cdot \ln(C_e) \quad (\text{Equation 2.10})$$

Where the symbol “ q_e ” represents the amount adsorbed per unit mass at equilibrium (mg/g); “ C_e ” represents the equilibrium concentration in solution (mg/L); “ AT ” represents the Temkin equilibrium binding constant related to maximum binding energy (L/g); “ B ” is same as “ RT/b ” and is equal to Temkin constant related to heat of adsorption (J/mol); “ R ” represents the universal gas constant (8.314 J/mol·K); “ T ” is the absolute temperature (K) while “ b ” is Temkin isotherm constant related to heat of sorption (J/mol). By plotting “ q_e ” against “ $\ln(C_e)$ ”, the slope and intercept of the graph, B and $B \cdot \ln(AT)$, are obtained and are used to calculate both the constants Temkin and Pyzhev (1940). In the Temkin adsorption model, parameters are

determined by plotting a graph of q_e (y-axis) vs $\ln(C_e)$ (x-axis). B is obtained from the slope, and $B \cdot \ln(AT)$ is obtained from the intercept (Xia *et al.*, 2022; Mudhoo, 2025).

2.23.4 Dubinin–Radushkevich (D–R) adsorption isotherm

The Dubinin-Radushkevich (D-R) isotherm, first developed by Chu *et al.* (2024) for modelling adsorption in microporous materials, was used. The Polanyi potential theory is the most basic adsorption theory, which assumes that the adsorption energy follows a Gaussian distribution on a non-uniform surface; the adsorption potential is the Polanyi potential (Xia *et al.*, 2022). The main advantage of the D–R model is that it can be used to distinguish between physical (physisorption) and chemical (chemisorption) adsorption mechanisms by calculating the mean free energy of adsorption, E , which is not possible with Langmuir and Freundlich models (Mudhoo, 2025). Dubinin–Radushkevich Linear Form (used in this study) is given by Equation 2.11 (Dehmani *et al.*, 2023).

$$\ln(q_e) = \ln(q_m) - \beta \cdot \varepsilon^2 \quad (\text{Equation 2.11})$$

In the above equation, the symbol " q_e " represents the amount adsorbed per unit mass at equilibrium (mg/g); q_m represents theoretical saturation capacity (mg/g); β represents the Dubinin–Radushkevich constant related to mean free energy of adsorption (mol^2/kJ^2); ε represents Polanyi potential, which is equal to $RT \cdot \ln(1 + 1/C_e)$. The mean free energy of adsorption, E , is calculated as shown by Equation 2.12 (Xia *et al.*, 2022; Dehmani *et al.*, 2023; Chu *et al.*, 2024).

$$E = 1 / \sqrt{2\beta} \quad (\text{Equation 2.12})$$

Mean free energy of adsorption is interpreted by Table 2.3 (Usgodaarachchi *et al.*, 2024; Xia *et al.*, 2022; Zaferani *et al.*, 2024)

Table 2.3: Interpretation of the mean free energy

E Value (kJ/mol)	Adsorption Type
$E < 8$	Physical adsorption (physisorption)
$8 \leq E \leq 16$	Chemical adsorption (chemisorption)
$E > 16$	Strong chemisorption/ion exchange

In Dubinin–Radushkevich adsorption isotherm, parameters are determined by plotting a graph of $\ln(q_e)$ (y-axis) against ε^2 (x-axis). The slope gives $-\beta$, and the intercept gives $\ln(q_m)$, from which E is computed (Dehmani *et al.*, 2023).

2.23.5 Comparative summary of isotherm models

Langmuir and Freundlich adsorption isotherms are most common models applied widely in adsorption studies. The two models differ significantly in almost all parameters. The comparison between the two models is summarized by the Table 2.4.

Table 2.4: Comparative summary of isotherm models

Parameter	Langmuir	Freundlich
Year introduced	1918	1906
Nature	Theoretical	Empirical
Surface type	Homogeneous	Heterogeneous
Adsorption layers	Monolayer only	Multilayer allowed
Maximum capacity	Defined (q_{max})	Not defined
Linear plot	C_e/q_e vs C_e	$\log q_e$ vs $\log C_e$
Key parameters	q_{max} , K_L	K_F , $1/n$
Best fit (this study)	Resorcinol ($R^2 = 0.9967$)	Phenol ($R^2 = 0.9816$)
Adsorption mechanism	Monolayer, uniform sites	Multilayer, mixed energies

The coefficient of determination (R^2) was used to assess the goodness of fit for each model. The model with the highest R^2 Value was taken as the best-fitting isotherm for each adsorbate. The equilibrium adsorption capacity (q_e) was calculated using the mass balance Equation 2.13.

$$q_e = (C_0 - C_e) \cdot V / m \quad (\text{Equation 2.13})$$

Equilibrium adsorption capacity is given by “ q_e ”. While “ C_0 ” is the initial concentration (mg/L); “ C_e ” is the equilibrium concentration (mg/L), V is the volume of solution (L), and “ m ” is the mass of adsorbent (g) (Xia *et al.*, 2022; Dehmani *et al.*, 2023; Chu *et al.*, 2024).

2.24 Research gap in the literature

Literature review has shown the need of water treatment. This is necessities by demand for clean and safe water for domestic use. Various methods of water treatment as discussed in this chapter have proofed to be efficient and reliable, however they fail to cater for elimination of residual aluminium ions from water treated using aluminium based coagulants. In addition, these methods do not remove phenolic compounds from water. As discussed earlier, exposure of aluminium ions and phenolic compounds to human has many detrimental effects on human health. This study investigates convenient methods of water remediation to eliminate these contaminants.

Available literature has reported that iron oxides nanoparticles are efficient adsorbents, however most of the methods used in their preparation as discussed earlier are not environmentally. This brings the need to study preparation of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using green synthesis. Green synthesis is known to be environmentally friendly since it does not use toxic chemicals and reagents unlike most chemical and physical methods of synthesis of nanoparticles. In the available literature, there is limited knowledge in use of pine apple (*Ananas comosus*) peels extract as capping agent in green synthesis of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles from iron(iii) salts. Therefore, this study explores new unbeaten paths.

2.25 Chapter summary

This chapter discussed the existing knowledge and brought out the research gap in water treatment. It discussed the effects of exposure of aluminium ions and phenolic compounds to human. The synthesis of nanoparticles and their characteristics were presented. Green synthesis of maghemite nanoparticles, their application in water remediation by adsorption and adsorption isotherms were also discussed in details.

CHAPTER THREE MATERIALS AND METHODS

3.1 Introduction

In this study, the main objective involved determination of residual aluminium ions from home-treated de-ionized and natural water samples using aluminium- based coagulant (Rock Alum). In addition, remediation of residual aluminium ions from treated water samples using maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles was determined. Also, application of maghemite ($\gamma\text{Fe}_2\text{O}_3$) in adsorption of phenolic compounds (phenol and resorcinol) from spiked water samples was investigated. Optimum conditions of adsorption were also studied.

3.2 Apparatus and reagents

Reagents and chemicals used included, Commercial stock solution of aluminium sulphate [$\text{Al}_2(\text{SO}_4)_3$], concentrated nitric(v) acid (HNO_3), iron (ii) sulphate (FeSO_4), iron(iii)chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$), sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), phenol ($\text{C}_6\text{H}_6\text{O}$), resorcinol ($\text{C}_6\text{H}_6\text{O}_2$) and concentrated hydrochloric acid (HCl). All chemical were analytical grade and were obtained from Sigma Aldrich Company, Kenya. All apparatus were washed using de-ionized water. Double distilled water was used to prepare spiked solutions.

3.3 Quantification of residual aluminium ions in water samples treated with water treatment coagulant (Rock Alum)

In this section, a study on quantities of residual aluminium ions on water samples treated with rock alum was carried out. Water treatment coagulants are alternative compounds used to treat water for domestic use especially in homes not served by commercial water treatment plants owned by local county governments. Most of these coagulants are aluminium based salts with simple procedure for their use. They are added into water without other post-treatment procedures carried out in conventional water treatment. Their main objective is to coagulate suspended and dissolved water impurities. This results in formation of the precipitates (flocs) which settles at the bottom of the tanks reducing turbidity of the water.

3.3.1 Standard solutions and validation of AAS

Standard solutions were prepared from double distilled water. Water sample were spiked with aluminium nitrate. Spiked standard solutions was prepared by dissolving dried high-purity salts of aluminium i.e. aluminium nitrate, in double distilled water. Aluminium solution with concentration 1000 ppm was prepared by dissolving 27.0 g of the salt in 1000 cm³ of double distilled water. Standard solutions were prepared by serial dilution of 1000 ppm solution to obtain working standard solutions of concentration of 10, 8, 6, 4 and 2 ppm. These solutions were used to calibrate the Atomic Absorption Spectrometer (AAS), Ice 3000 series made by Thermo-fisher from German. The calibration curves obtained from spiked solutions was used to calculate the recoveries.

3.3.2 Home water treatment coagulants

Home water treatment coagulants (Rock Alum) were bought from local chemical shops in around Thika and Nairobi, Kenya. These water treatment compounds were classified and labeled based on the manufacturer as shown in Table 3.1

Table 3.1: Different brands of coagulants used based on manufacturer

Brand name	Active compound	Manufacturer	No. of samples
ROCK ALUM-1	Aluminium sulphate (Al ₂ [SO ₄] ₃)	Jolichem Suppliers Ltd. Nairobi Kenya	3
ROCK ALUM-2	Aluminium sulphate (Al ₂ [SO ₄] ₃)	Supreme Farmcare Ltd. Nairobi Kenya	3
ROCK ALUM-3	Aluminium sulphate (Al ₂ [SO ₄] ₃)	Impact Chemicals Ltd. Nairobi Kenya	3
ROCK ALUM-4	Aluminium sulphate (Al ₂ [SO ₄] ₃)	Avachem Nairobi Kenya	3

3.3.3 Treatment of de-ionized water samples

De-ionized treated water sample was prepared by accurately weighing 0.125 g of coagulant from each manufacturer at a time using an analytical balance and adding it to 500 cm³ of de-ionized water. The solution was stirred using a magnetic stirrer for 30 minutes. It was then allowed to stand for 24 hours to enable coagulation and

settlement of the flocs. The water sample was filtered using Whatman filter paper no. 40 (8 μm particle retention size) and labelled as sample A.

Solution B was prepared similarly by dissolving 0.025 g of coagulant from the same manufacturer in 500 cm^3 of de-ionized water and procedure above repeated. De-ionized water sample C was prepared similarly but 0.0125 g of coagulant was used to treat 500 cm^3 . Sample D was the control sample where 0.0 g (Zero) coagulant was added in solution D, as shown, Table 3.2 (Hasan, 2023; Zwane *et al.*, 2024).

Table 3.2: Different masses of coagulants used to treat de-ionized water samples

Treated water samples	Mass of coagulant used (g)	Vol of de-ionized water used (cm^3)
A	0.1250	500
B	0.0250	500
C	0.0125	500
D	0.0000	500

After treatment the water samples were quantitatively analyzed for residual aluminium ions using Atomic Absorption Spectrometer (AAS). And the data obtained statistically analyzed. This was done to determine the residual aluminium ion obtained for each coagulant used in the study.

3.3.4 Treatment of natural water.

Homogenous coagulant mixture was obtained by mixing equal masses of all the four different coagulants. This was done by accurately weighing 4 g of each coagulant, mixing them and grinding in a mortar using pestle to form 16 g of Homogenous coagulant mixture used in treatment of natural water samples. Water samples were collected from 3 different rivers labelled River-1(Chania), River-2(Thika) and River-3 (Maragwa). These rivers were chosen since they are some of the main rivers in central Kenya, therefore they represent average river water samples ideal for this study. Water samples were also collected from 3 different boreholes, labelled Borehole-1(Juja), Borehole-2(Thika), and Borehole-3(Maragwa). These boreholes are distributed along central Kenya. The natural water samples were collected from rivers and boreholes in central Kenya as shown by Figure 3.1.

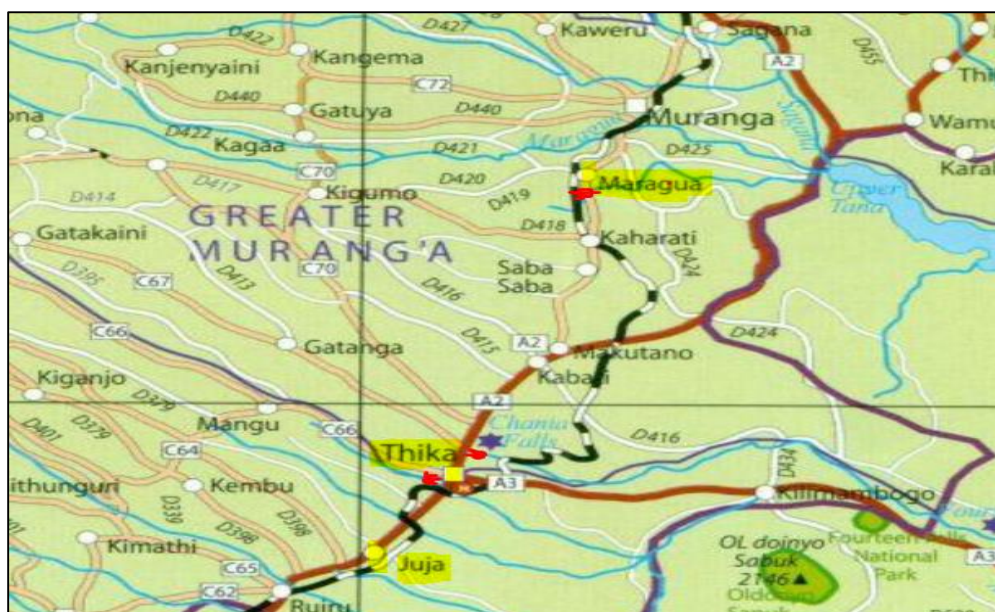


Figure 3.1: Water sampling sites. Red dot shows river water sampling site (Chania, Thika and Maragwa rivers from bottom respectively); Yellow highlight shows borehole water sampling sites (Juja, Thika and Maragwa respectively)

The natural water samples were treated with the Homogenous coagulant mixture prepared earlier. The mass of the Homogenous coagulant mixture used in water treatment was varied from 0.1250, 0.0250, 0.0125 and 0.0000 g each in 500 cm³ of natural water sample. The mixture was stirred using magnetic stirrer at medium revolutions for 20 minutes. It was then allowed to stand for coagulation to occur for 1 hour. The treated water samples were filtered using Whatman filter paper no. 40 (8µm particle retention size) and placed in labelled 25 ml volumetric flasks using the codes in the Table 3.2. The samples were then analyzed using AAS to determine the average concentration of residual in triplicate (Gandhi *et al.*, 2022; Idris *et al.*, 2021).

3.3.5 Quantification of residual aluminium ions in water samples before adsorption

De-ionized and natural water samples treated with coagulant (Rock Alum) as shown by procedure 3.3. The samples were analyzed for residual aluminium ions by Atomic Absorption Spectrometer (AAS). For background correction, three blanks were pre-tested samples for residual metal ion. The data obtained was analyzed in reference to data obtained after adsorption (Abrham & Gholap, 2021; Kassim *et al.*, 2022).

3.4 Synthesis and characterization of nanoparticles

Maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles were prepared by reduction FeCl_3 by green synthesis using pineapple peels extract as the capping agent. The resulting solution was precipitated using sodium hydroxide to form $\text{Fe}(\text{OH})_3$. After 3 washing using diluted ethanol, the precipitates were dried in the oven at 100°C till they are brittle. The precipitates were calcinated at variable temperature to obtain maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles.

3.4.1 Preparation of Pineapple (*Ananas comosus*) peels extract

The pineapple (*Ananas comosus*) peel extract is the material of choice to act as capping agent in reduction of Fe^{+3} ions. This is because it contains high phenolic antioxidants necessary for the reduction of iron salts (Ketnawa *et al.*, 2022; Ramos *et al.*, 2024). Pineapple peels were obtained from fruit markets in Thika town. About 500 g of pineapple peels were washed in tap water. Inner fruit remains were scrapped off and then washed again in de-ionized water. The peels were shredded into small pieces using kitchen knife. The pieces were sun dried for about 5 days until they were hard and brittle. The shredded dry peels were rinsed in de-ionized water to remove any dust acquired in process of sun drying. Peels were then dried further in an oven at 80°C for about 4 hours. Heavy duty blending mill Signature professional blender model SG-BL07PP was used to grind the dry peels into fine powder then stored in a dry air tight clean plastic container.

About 30 g of the peel powder were weighed and added into 1000 cm^3 of de-ionized water. The suspension was heated in a magnetic stirrer at 70°C for 1 hour while stirring at medium speed of about 100 rpm as shown by Figure 3.3. The peel extract was then allowed to settle and cool for about 2 hours. The mixture was decanted into a clean beaker before double filtration in series using Whatman filter paper no.42 ($2.5\text{ }\mu\text{m}$ particle retention). The pineapple peels extract obtained was stored in a labeled sealed 500 ml volumetric flask at room temperature ($24\text{-}26^\circ\text{C}$).



Figure 3.2: Preparation of pineapple peels extract

Iron (iii)chloride solution (0.5M) was used as the precursor solution. It was prepared by accurately measuring 20.275 g of analytical grade iron (iii) chloride and adding it into 250 cm³ de-ionized water in a clean glass beaker. The solution as stirred by a magnetic stirrer for 1 hour. The solution was filtered using Whatman filter paper no.40 (8 µm particle retention size) then labelled as Fe⁺³ precursor solution. It was stored in a stoppered volumetric flask (Bhuiyan *et al.*, 2020; Aida *et al.*, 2023; Ekwumemgbo *et al.*, 2023).

3.4.2 Green synthesis of maghemite ($\gamma\text{Fe}_2\text{O}_3$) using pineapple (*Ananas comosus*) peels extract

A volume of 50 cm³ of 0.5 M iron (iii) chloride solution was placed into a 500 cm³ beaker on a magnetic stirrer. An equal volume of pineapple peel extract was added in portions of 5 cm³ at a time from the burette with constant stirring for 1 hour at room temperature (24-26 °C). Then 0.5 M NaOH solution was added from burette to the mixture dropwise until the pH was 11. The pH was adjusted using 0.5 M HNO₃ to stabilize pH at 11. The solution changed colour to a dark brown precipitate due to formation of iron(iii)hydroxide. The mixture was stirred for about 30 minutes in magnetic stirrer at moderate speed. The mixture was then allowed to settle for 24 hours.

After overnight stay, the brown precipitates settled at the bottom of the beaker. The upper light brown liquor was carefully decanted off. The precipitates were washed and rinsed with a mixture of de-ionized water and absolute ethanol at ratio of 1:1 to remove traces of precursor solutions. After washing, the mixture was allowed to stay for 24 hours for precipitates to settle. This washing procedure was repeated 3 times before drying in an oven at 100 °C for 10 hours until it formed brown brittle residue. The residue was transferred to different crucibles and calcinated in a furnace at different temperatures as show in Table 3.3.

Table 3.3: Maghemite samples at different calcination temperatures

Sample	Code	Calcination temperature (°C)
1	P1A	350(No capping agent)
2	P2C	0 (Not calcinated)
3	P2D	250
4	P2E	300
5	P2F	350
6	P2G	400
7	P2H	450

Calcination was done at variable temperature for 2 hours for each sample as shown by Figure 3.3 that illustrated loading of the furnace with the samples for calcination. Control samples (P1A) were prepared by calcinating Fe(OH)₃ prepared without using capping agent (pineapple peels extract) before calcination at 350 °C (Gutierrez *et al.*, 2024).



Figure 3.3: Calcination of the $\text{Fe}(\text{OH})_3$ to form Maghemite ($\gamma\text{Fe}_2\text{O}_3$)

The nanoparticles were stored in labelled stoppered sample vials ready for characterization and adsorption studies (Aida *et al.*, 2023; Bhuiyan *et al.*, 2020).

3.4.3 Characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using XRD

Sample maghemite nanoparticles were dried in an oven for 12 hours at 80°C . Ground samples were transferred into appropriately labeled glass bottles. The powder of the solid analyte was filled into an empty sample holder from the sample vial. Using a glass slide, the analyte powder was then gently pressed. The powder surface was smoothened. Carefully the excess powder from the sample holder edges was scrapped off. The sample holder was carefully installed into the appropriate XRD analyte slot. The sample was analyzed at pitch angle of 2θ , Resolution of 4096, incident angle 14° at wavelength of $1.54056\text{\AA}$ and passing time of 60 seconds (Bilardo *et al.*, 2022; Falke *et al.*, 2024; Ramos-Guivar *et al.*, 2024).

3.4.4 Characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using FTIR

Fourier transform infra-red spectroscopy (FTIR) is an effective analytical technique for surface analysis. It identifies the main functional and chemical bond in a compound. Maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles were dried in an oven for 2 hours at 100°C (Durak & Depciuch, 2020).

The surface of maghemite was analyzed before and after adsorption. The FTIR spectrometer was allowed to warm up for about 20 minutes before calibration using polystyrene standard sample. Full range scan was done between 400-4000 nm wave number. The mode of analysis was, transmittance. This analysis was done to characterize the surface of nanoparticles and also to confirm the phenolic groups bind to the adsorbent to ascertain the success of the removal of phenolic groups from water. The detection of functional groups on a sample was confirmed by the manifestation of differentiated peaks at specific wavelengths (Eid, 2022; Pasieczna-Patkowska *et al.*, 2025).

3.4.5 Characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using UV-Vis

The maghemite nanoparticles was cleaned in absolute ethanol then rinsed with de-ionized water. Nanoparticles were dispersed in de-ionized water to avoid aggregation. The mixture was diluted farther to the desirable concentration in a sample cuvette. De-ionized water was used as the blank. Then placed in sample slot before analysis. The sample was scanned at full wavelength at between 190 nm-1100 nm (Xu *et al.*, 2022; Sudharshan & Swetha, 2023; Vogt *et al.*, 2023).

3.5 Adsorption of residual aluminium ion using ($\gamma\text{Fe}_2\text{O}_3$) maghemite nanoparticles

In this study, the batch adsorption procedure applied was used in previous studies by Murage *et al.*, (2022) and Sosun *et al.*, (2022). During adsorption, water samples whose concentration of aluminium ions had been predetermined in section 3.3 was used. Batch adsorption was done in triplicates. A volume of 25 cm³ of the analyte was transferred into a 50 ml conical flask. A mass of accurately measured 0.5 g of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles adsorbent was placed into the sample analyte. The mixture was stirred by magnetic stirrer at 1000 rpm for 30 minutes.

After adsorption the mixture was decanted to separate the suspension. It was then filtrated using Whatman filter paper no. 40. After filtration, the filtrate was labeled based on the different variables applied awaiting analysis of concentration of residual aluminium and phenolic compounds concentration. The adsorption was repeated at pH of 4, 5, 6, 7, 8 and 9. Adsorption was also carried out at variable temperature at 35, 45, 55, 65 and at 75⁰C. Contact time was also varied at 15, 30, 45, 60 and 75 minutes.

The water samples after adsorption were analyzed for residual aluminium ion using AAS. The quantity of the metal ions adsorbed was calculated by the formula used by the author (El Hosry *et al.*, 2023; Elkhatat *et al.*, 2021; Kassim *et al.*, 2022; Sosun *et al.*, 2022).

$$\% \text{ Adsorption} = \frac{(C_i - C_f) \times 100}{C_i} \quad (\text{Equation 3.1})$$

Where C_i = initial concentration the analyte before adsorption

C_f = final concentration of the analyte after adsorption.

3.6 Adsorption of phenol and resorcinol (Phenolic compounds) using maghemite

Phenolic compounds are persistent water pollutants commonly found in natural water as discusses in section 2.7. In this study, effectiveness of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles in remediation of phenolic compounds from water was investigated. Effect of change in pH, temperature and contact time was studied. Spiked solutions of phenol and resorcinol which are some of the most common phenolic compounds and also water pollutants were used in this study.

3.6.1 Preparation of phenol solution (Water samples spiked with phenol)

Using analytical balance, 5.0000 g of phenol was weighed and dried in an oven at 80 °C for 2 hours. Phenol solution was prepared by dissolving 0.1000 g of dried phenol crystals into about 200 cm³ de-ionized water in a 1000 mL volumetric flask. The solution was shaken before topping up to the mark. It was labelled 100 ppm phenol stock solution.

Phenol standard solutions were prepared by serial dilution of stock solution from 100 ppm. The concentration of standard solutions prepared were 20, 40, 60, 80, and 100 ppm. The standard solutions were used to calibrate UV-Vis. Calibration curves were drawn using absorbance against concentrations of the standard solutions. Calibration of equipment was done to ensure sensitivity and accuracy of data obtained during analysis. Data obtained after determination of concentration of phenolic compounds in spiked water samples, before adsorption and after adsorption was used to determine effectiveness of adsorption process (Himmi *et al.*, 2022; Giglio *et al.*, 2023; Yusoff, 2023).

3.6.2 Preparation of resorcinol solution (Water samples spiked with resorcinol)

Using analytical balance 3.0000 g of resorcinol was weighed and dried in an oven at 80 °C for 2 hours. Phenol solution was prepared by dissolving 0.0999 g accurately weighed of dried resorcinol crystals into about 200 cm³ de-ionized water in a 1000 mL volumetric flask. The solution was shaken to form homogeneous solution before topping up to the mark. It was labelled 100 ppm resorcinol stock solution. Standard solutions for resorcinol were prepared by serial dilution of stock solution from 100 ppm. The concentration of standard solutions prepared were 100, 80, 60, 40 and 20 ppm (Himmi *et al.*, 2022; Giglio *et al.*, 2023; Yusoff, 2023).

3.6.3 Adsorption of phenolic compounds (phenol and resorcinol) using maghemite

Batch adsorption analysis was done by accurately measuring 50 cm³ of 100 ppm phenol or resorcinol solution into a 100 cm³ glass beaker. Using analytical weighing machine, 0.5 g of maghemite ($\gamma\text{Fe}_2\text{O}_3$) was measured and added into sample solution. The mixture was stirred using magnetic stirrer before filtration using Whatman filter paper no.40. Batch adsorption was carried out by varying physical conditions. That is, pH and temperature and contact time. The pH was varied from 4, 5, 6, 7, 8 and 9. This was done at room temperature of 25 °C. The adsorption was repeated at variable temperature of 35, 45, 55, 65 and 75 °C. The contact time was varied from 15, 30, 45, 60, and 75 minutes at room temperature of 25 °C.

3.7 Adsorption at optimum conditions

The study was enhanced further by repeating the adsorption procedure as described in chapter 3.5 (Adsorption of residual aluminium ions) and chapter 3.6.3 (Adsorption of phenolic compounds) from spiked solutions from de-ionized water. This was repeated at optimum conditions of pH, temperature and contact time from results obtained in procedure 3.5 and 3.6.3. This was done to determine the effectiveness of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles in adsorption of in the removal of both aluminium and phenolic compounds from water samples at optimum conditions.

3.8 Data analysis

The data obtained from analysis of water samples using AAS (residual aluminium) and UV-Vis (residual phenolic compounds) before and after adsorption was used to determine percentage adsorption. The percent adsorption was calculated from the data

obtained. To avoid type 1 or 2 errors in the statistical work, analysis of variance was determined using statistical analysis of variance (ANOVA). The percentage adsorption and other statistical analysis was done at $P=0.01$ (1%) significant level of confidence was calculated using the SPSS statistical software.

3.9 Chapter summary

In this, water samples (De-ionized and nature) were treated using aluminium based coagulants (Rock Alum), then filtrate analyzed for residual aluminium ions using AAS. Maghemite nanoparticles were synthesized by green method from iron (iii) chloride which was capped using pineapple (*Ananas comosus*) peels extract. The nanoparticles were characterized using XRD, FTIR and UV-Vis. Batch adsorption of residual aluminium from de-ionized and natural water samples was carried out using 0.5 g of maghemite. Adsorption was done varying conditions of pH, contact time and temperature. Batch adsorption of phenolic (phenol and resorcinol) compounds was done from spiked water samples at varying conditions of pH, contact time and temperature using 0.5 g of maghemite. The adsorption of residual aluminium and phenolic compounds was repeated at optimum.

CHAPTER FOUR RESULTS AND DISCUSSION

4.1 Introduction

This chapter discusses both experimental and statistical data obtained in this study. This work involved synthesis of ($\gamma\text{Fe}_2\text{O}_3$) maghemite nanoparticles by green method using pineapple peels extract. From the study, it was found that variation in synthesis conditions had a great effect on the characteristics of the nanoparticles obtained. The nanoparticles formed were characterized using XRD and FTIR and different patterns obtained from analysis were discussed in this chapter.

Water samples were treated using aluminium based coagulants samples (Rock Alum). The results of residual aluminium ions obtained are presented and discussed in this chapter. Then adsorption of residual aluminium and phenolic compounds from de-ionized and natural water samples was carried out. The results and discussion on effects on change in adsorption conditions are also discussed. The study found that water treated had high residual aluminium ions. This study also reported that adsorption of aluminium ions was best at pH of 6 and reduced significantly with decrease in pH. Change in adsorption temperature had no significant effect on percentage adsorption.

4.2 Quantification of residual aluminium ions in water samples treated with home water treatment coagulant (Rock Alum)

In this section, the results on residual aluminium ions are discussed. These results were obtained from Atomic Absorption Spectrometer (AAS) on analysis of water samples after treatment with Rock Alum. The AAS was calibrated using standard solution before using it in quantification of residual aluminium ions. De-ionized water samples and natural samples (river and borehole water samples). Treatment was done with variable masses of coagulant as described in section 3.3. The results on the residual aluminium ions are presented in form of tables and graphs and discussed in details.

4.2.1 Atomic Absorption Spectrometer (AAS) validation

Standard solutions of analytical grade aluminium nitrate were prepared using de-ionized water by serial dilution. Standard solutions of concentration of 2, 4, 6, 8 and 10 ppm were used to calibrate Atomic Absorption Spectrometer (AAS). On analysis,

absorbance readings were as shown by the Table 4.1. In the study, the tests were done in triplicate (N=3) and values presented in the table as Mean $\pm$ SD.

Table 4.1: Calibration of AAS

Concentration (ppm)	Absorbance	p-value (t-test)
2	0.0010 $\pm$ 0.0000 ^a	0.001
4	0.0020 $\pm$ 0.0001 ^b	<0.001
6	0.0040 $\pm$ 0.0001 ^c	<0.001
8	0.0050 $\pm$ 0.0001 ^d	<0.001
10	0.0070 $\pm$ 0.0002 ^e	<0.001
p-value (ANOVA)	<0.001	

In Table 4.1, the superscript letters (a, b, c, d, e) are statistical comparison markers. Each letter indicates that the value in that row is statistically significantly different from the values carrying a different letter. Values sharing the same letter would be considered not significantly different from each other. Since all five absorbance readings carry different letters (a through e), it means every concentration level produced a statistically distinct absorbance reading, that is, none of them were similar to each other. In the context of this AAS calibration table, the letters confirm that as concentration increases (2, 4, 6, 8, 10 ppm), the absorbance values are all meaningfully and statistically different from one another, indicating it is a good calibration curve which gave a clear and distinguishable response at each concentration level.

The calibration curve was drawn as a linear plot of $y=0.000750x+(-0.000700)$ over the concentration of standards ranging from 2.0- 10 ppm with a correlation coefficient (R^2)= 0.986842 as shown in Figure 4.1

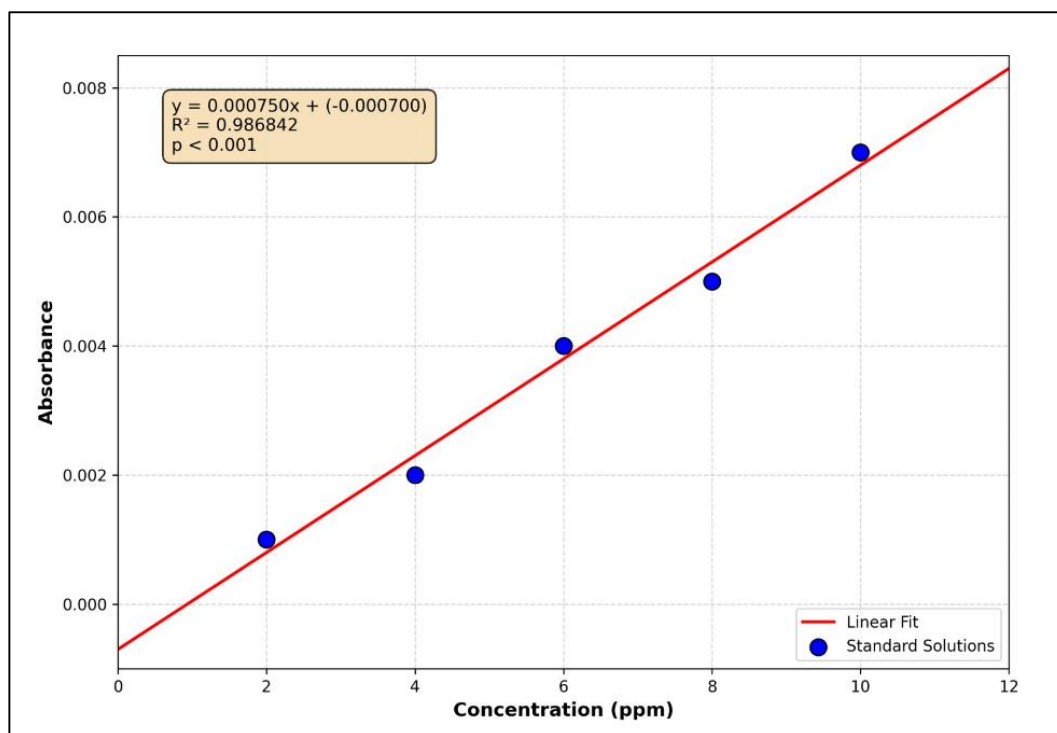


Figure 4.1: Calibration curve of AAS

4.2.2 Residual aluminium ions in treated de-ionized water samples

De-ionized water samples were treated using coagulants (Rock Alum) from different manufacturers as shown in Table 3.1. as described in section 3.3. Varying masses of coagulant were used as shown in Table 3.2. The treated water samples were analyzed using Atomic Absorption Spectrometer (AAS).

The results of an analysis for residual aluminium ions in treated de-ionized water showed high residual aluminium ions. Coagulant-3 gave the relatively highest concentration of residual ions to a maximum of 15.01 ppm when a mass of 0.125 g was used in 500 cm³ of de-ionized water. Coagulant-1 gives the lowest residual ions (7.41 ppm) for the same volume of water. It was observed that when Zero mass of coagulant was used, the water samples exhibited untraceable concentration of residual aluminium ions. This observation explains that the residual ions are attributed to using coagulants in the water treatment. The higher the mass of coagulants used, the higher the aluminium concentration, as shown in the Table 4.2.

Table 4.2: Concentrations of residual Al³⁺ ions in treated de-ionized water

Sample	Mass of Coagulant (g)	Residual Al ³⁺ concentrations (ppm)				
		Coagulant-1	Coagulant-2	Coagulant-3	Coagulant-4	Average
A	0.1250	7.41±0.15 ^a	11.39±0.23 ^b	15.01±0.31 ^c	8.99±0.18 ^a	10.7
B	0.0250	2.36±0.05 ^a	4.06±0.08 ^b	4.83±0.10 ^c	3.47±0.07 ^b	3.68
C	0.0125	1.90±0.04 ^a	2.14±0.04 ^a	3.65±0.07 ^b	2.95±0.06 ^b	2.66
D	0.0000	0.00±0.00 ^a	0.00±0.00 ^a	0.00±0.00 ^a	0.00±0.00 ^a	0.00
p-value	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

In Table 4.2, the superscript letters (a, b and c) are statistical comparison markers. For example, coagulant-1 and coagulant-4 both marked with superscript “a” are not significantly different from each other. Coagulant-2 marked by superscript “b” is significantly different from both Coagulant-1 and Coagulant-4. Coagulant-3 marked superscript “c” is significantly different from all others. The p-value < 0.001 indicates these differences are statistically significant.

On average, Table 4.2 shows concentrations of residual ions were higher than the tolerable recommended levels of 0.1mg/L (0.1 ppm) by the World Health Organisation (Edition, 2011). Coagulant-1, which had the lowest residual ions, gave a value of 1.90 ppm, with the lowest mass of 0.0125 g of coagulant used in 500 cm³ of de-ionized water. These elevated levels of aluminium pose a significant health concern to water consumers. This presents the need to address use of home water treatment coagulants (Rock Alum) in order to control human exposure to residual aluminium ions, resulting in aluminium poisoning.

4.2.3 Residual aluminium ions in treated natural water

The Homogenous coagulant mixture prepared by mixing all the four coagulants was used in treatment of natural water samples. Natural water samples were collected from 3 different rivers and 3 boreholes within central Kenya. After treatment, coagulation and filtration, the water samples were analyzed for residual aluminium using Atomic Absorption spectrometer (AAS).

The results obtained demonstrated that the river water samples, on average, gave lower residual aluminium ions than treated de-ionized water treated with equal mass of coagulant. Water samples from Thika and Chania river exhibited higher residual aluminium. When 0.125 g of coagulant were used in 500 cm³ of water they give 4.68 and 5.73 ppm respectively while Maragwa river gave 2.28 ppm. These observations were attributed to human activities along the two rivers. Thika and Chania rivers pass through areas with high number of large-scale coffee and flower farms. It has been reported that these farms contribute to water pollution due uncontrolled use of farm chemicals which results to elevated aluminium content in river water (Murage *et al.*, 2022; Ali *et al.*, 2025).

When 500 cm³ of river and borehole water samples were treated with 0.125 g of Homogenous coagulant mixture, the results gave average of 4.23 ppm and 3.47 ppm of the residual aluminium ions respectively. These results were lower than 8.99 ppm for de-ionized water treated with similar mass of coagulant. This was attributed to the lack of suspended particles which causes turbidity in de-ionized water. Therefore, negligible quantities were used to form the flocs, resulting in a high concentration of residual metal ions. In Figure 4.2, river water samples formed flocs when treated with the coagulant. These flocs appeared as dirty white residues in the beaker on the right (Figure 4.2).



Figure 4. 2: Image showing flocs in the treated river water samples

The study observed that flocs are formed from the settlement of suspended particles in the natural water, while de-ionized water had none. Therefore, when equal volumes of natural water and de-ionized water samples were treated with an equivalent mass of coagulants, natural water gave lower residual aluminium than de-ionized water, as shown in Table 4.3 and Table 4.4 of residual aluminium in river water and borehole water.

Table 4.3: Concentrations of residual Al^{3+} ions in treated river water

Sample	Statistics	Mass of Coagulant (g)	Residual Al^{3+} concentrations (ppm)			
			River 1	River 2	River 3	Average
A	N=3	0.1250	4.68±0.09 ^a	5.73±0.11 ^b	2.28±0.05 ^c	4.23±0.89
B	N=3	0.0250	1.04±0.02 ^a	1.69±0.03 ^b	1.43±0.03 ^b	1.39±0.17
C	N=3	0.0125	0.56±0.01 ^a	0.97±0.02 ^b	1.51±0.03 ^c	1.01±0.24
D (Control)	N=3	0.0000	0.98±0.02 ^a	0.05±0.001 ^b	0.60±0.01 ^c	0.54±0.24
p-value	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

Table 4.4: Concentrations of residual Al³⁺ ions in treated borehole water

Sample	Statistics	Mass of Coagulant (g)	Residual Al ³⁺ concentrations (ppm)			
			Borehole 1	Borehole 2	Borehole 3	Average
A	N=3	0.1250	4.06±0.08 ^a	3.33±0.07 ^b	3.02±0.06 ^b	3.47±0.28
B	N=3	0.0250	2.03±0.04 ^a	1.06±0.02 ^b	0.98±0.02 ^b	1.36±0.31
C	N=3	0.0125	0.25±0.005 ^a	0.84±0.02 ^b	1.01±0.02 ^c	0.70±0.21
D (Control)	N=3	0.0000	0.20±0.004 ^a	0.01±0.0002 ^b	0.00±0.00 ^b	0.07±0.06
p-value	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

In Tables 4.3 and 4.4, same superscript letters indicated there is no significant difference in values. That is, values sharing the same superscript letter (for example, two values marked with 'a') are statistically similar to each other. Different superscript letters indicate the values are significant different. That is values with different superscript letters (for example, one marked 'a', another marked 'b') are statistically different from each other.

On average river water gave higher concentrations of residual aluminium ions than borehole water. When 0.125 g of coagulant was used in 500 cm³ of water samples, river water samples gave average of 4.23 ppm while borehole gave 3.47 ppm of residual aluminium ions. When 0.0125 g of coagulant was used river and borehole water samples gave an average of 1.01 and 0.070 ppm respectively as shown in Table 4.5.

Table 4.5: Overall comparison of residual Al³⁺ across the three water types

Water Type	Statistics	Mean residual Al ³⁺ ±SE (ppm) at different coagulant doses		
		0.1250g/500mL	0.0250g/500mL	0.0125g/500mL
De-ionized water	N=9	10.70±1.52 ^a	3.68±0.50 ^b	2.66±0.36 ^c
River water	N=9	4.23±0.89 ^a	1.39±0.17 ^b	1.01±0.24 ^b
Borehole water	N=9	3.47±0.28 ^a	1.36±0.31 ^b	0.70±0.21 ^c
p-value	<0.001	<0.001	<0.001	<0.001

Treated river water samples exhibited higher concentrations of aluminium ions than treated borehole water. Results of analysis of the control samples (Zero coagulant added) indicated concentration of river water and borehole been 0.54 and 0.07 ppm respectively. This may be attributed to human activities such as release of farm and industrial wastes to water natural water sources resulting in elevated residual ions in untreated water samples.

A study carried by Angelova *et al.*, (2020) on determination of aluminium ions in Iskar rivers in Bulgaria reported that their waters contained between 0.148- 0.199 ppm of metal ions. This concentration was higher than recommended maximum limit of aluminium in drinking water by environmental quality standards of Bulgaria to a maximum of 0.3 ppm. High aluminium content in the river water was also attributed to the effect of the mineral and chemical composition of the soil in the locality (Angelova *et al.*, 2020).

In a study carried out on aluminium content in natural waters and soils in turkey, reported that most aluminium in natural water is as a result of interaction of water with rocks. In Canakkale-kirazli region in turkey, most rocks were found to contain about 7.8 to 8.2 % aluminium. This contributed to some natural waters having sometimes more than 100 mg/L (100 ppm) aluminium content especially in acidic waters. But the aluminium concentration reduced with increase in pH due to low solubility especially at pH range of 6-8 (KOBYA, 2023).

A study by Ahmed *et al.*, (2019) analyzed residual aluminium ions in treated water samples from different areas in Malaysia. Water samples were collected from 8 sampling stations from other water treatment plants. They reported that the samples had high residual aluminium ions. It was observed that the concentration increased with an increase in water acidity, while near-neutral water samples exhibited the lowest concentration. The aluminium concentration ranged from 0.001 to 8.00 ppm. These levels were higher than the permissible Malaysian legal limits of 0.2 ppm. The residual aluminium ions were attributed to using aluminium-based water coagulants in water treatments (Ahmed *et al.*, 2019).

Previous studies have reported that natural raw water has lower aluminium content than treated water. This observation was similar to a survey conducted by Krupińska (2020) on the effects of using aluminium-based coagulants on the concentration of residual aluminium ions in treated water reported that untreated water had a concentration range of 0.0001 and 1.0 ppm. However, the aluminium content increased with decreased pH, especially lower than 5. This is attributed to the low solubility of aluminium in water at a pH of 6-8. Therefore, aluminium content is generally lower in natural surface water where the pH is about 7. The aluminium in natural water results from water interacting with naturally occurring aluminium salts in the soil. The study found that tap water contained elevated metal ions, attributed to using aluminium salts as coagulants. Aluminium sulphate, when used in water treatment, increases the dissolved aluminium in water due to its lower molecular weight than poly aluminium coagulants (Krupińska, 2020).

Coagulants are used in water treatment to remove microorganisms, organic matter, total suspended solids (TSS), COD and other solid contaminants, eliminating water turbidity (Nimesha *et al.*, 2022). However, this treatment results in high aluminium content, making tap water unsafe for human consumption due to various health challenges related to aluminium poisoning (Farhat & Ali, 2020). Consumption of contaminated water results in compromised central nervous system, Alzheimer's disease, Renal failure, bone defects, among others (Murage & Waithaka, 2025). The total exposure to aluminium via consumption depends on the method of water treatment and the specific coagulant used. The situation is aggravated in areas where

treated water already has high natural aluminium content from physicochemical and mineralogical sources (Tahraoui *et al.*, 2024).

Previous studies have reported that the amount of residual aluminium ions in treated water depends on physical factors such as the amount of coagulant used, coagulation temperature, pH, and turbidity of the treated water (Wallace *et al.*, 2023). Most developed countries, such as France, Poland, Sweden, Japan, and the United States of America, have set maximum limits for the aluminium content in drinking water to be less than 0.1 ppm. These limits are set to ensure the safety of the consumers (Krupińska, 2020).

4.3 Synthesis and characterization of maghemite($\gamma\text{Fe}_2\text{O}_3$) nanoparticles

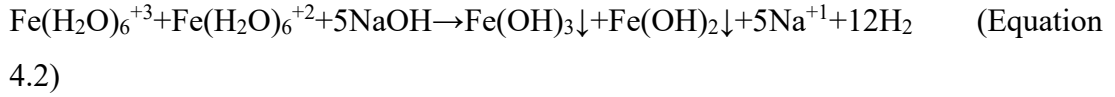
Maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles were prepared by green synthesis. Iron(iii)chloride was used as precursor solution, which was reduced by pineapple (*Ananas comosus*) that acted also as a capping agent. Sodium hydroxide was used to iron(iii)hydroxide which was calcinated at different temperature. Nanoparticles formed were characterized using XRD, UV-Vis and FTIR. Two control samples were prepared. First control sample was prepared without using a capping agent and calcinated at 350 °C, while the second one was prepared without calcinating.

4.3.1 Green synthesis of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles

Iron (iii) chloride was used as precursor solution. In solution hydrated iron (iii) chloride exists in form of $\text{Fe}(\text{H}_2\text{O})_6^{+3}$. Pineapple peels extract was used as reducing (capping) agent. The phenolic functional groups present in the pineapple (*Ananas comosus*) peels extract reduces iron (iii) ions to iron (ii) ions as shown in Equation 4.1 (Abdullah *et al.*, 2023).



However, some of the Fe^{+3} remains unreduced making the solution to be predominantly brown. Addition of sodium hydroxide to the solution results in precipitation of iron (iii) hydroxide. This occurred at high alkaline environment of pH values greater than 10 (Ramos-Guivar *et al.*, 2024)



Iron (iii) hydroxide precipitate on drying at 100 °C and calcination at 450 °C, where it decomposes to form magnetite (Fe₃O₄).



On further calcination at extended time in presence of oxygen, magnetite is oxidized forming maghemite nanoparticles as shown in Equation 4.4 (Abdullah *et al.*, 2023; Ramos-Guivar *et al.*, 2024).



4.3.2 Characterization of maghemite($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using XRD

The XRD patterns presented in Figures 4.3 depict the diffraction profiles of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles recorded in the range of 28-65° using EQUINOX 100 XRD spectrometer. The samples exhibited different characteristics at different calcination temperature (250, 300, 350, 400 and 500 °C). As observed from Figure 4.3, the diffraction pattern exhibits distinct peaks at 30.2°, 34.5°, 43.2°, 53.5°, 57.2°, and 62.8°, corresponding to the (220), (311), (400), (422), (511), and (440) crystal planes respectively. A study by Kim *et al.*, (2012) provided similar results, indicating that the diffraction peaks were indexed to crystallite cubic maghemite structure that matched with the available Joint Committee on Powder Diffraction Standards (JCPDS 39-1346). The effects of calcinating temperature were examined by monitoring the peak position.

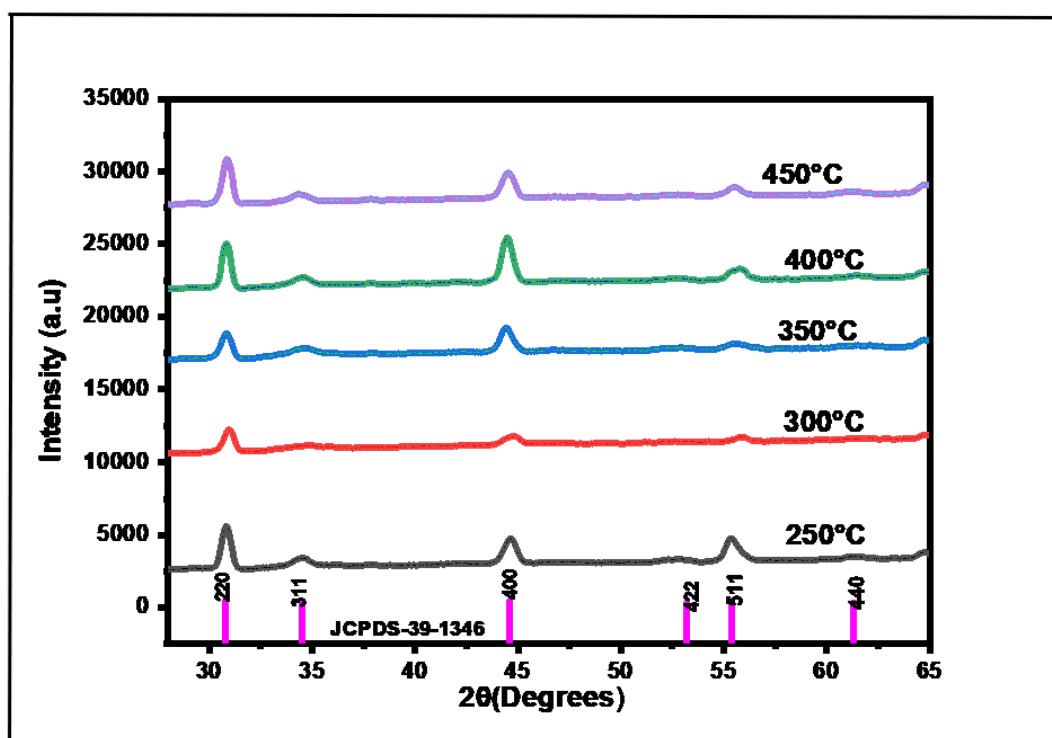


Figure 4.3: XRD Pattern for maghemite nanoparticles at varying calcination temperature

No impurity peaks were observed indicating that the synthesized material was of good crystallinity. This was also confirmed by the reduced full width at half maximum (FWHM) and higher XRD peak intensities of nanoparticles as shown by analysis of 220 plane in Figure 4.4.

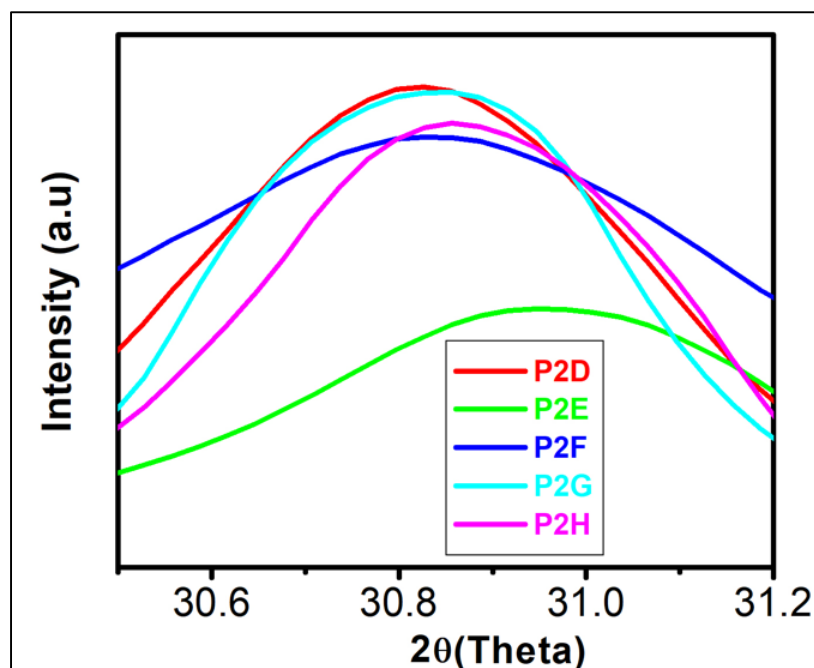


Figure 4.4: XRD Pattern Showing Magnified (220) Peak for the Synthesized maghemite different calcination temperature

The XRD pattern of plane (220) was used to determine the effects of calcinating temperature on shift position and intensity. The plane was used because it had the highest peak as compared to all the other nanoparticles. It was noted that there was variation in the FWHM and peak intensities when calcinating temperature was varied. Further analysis was conducted and the results represented in Figure 4.5.

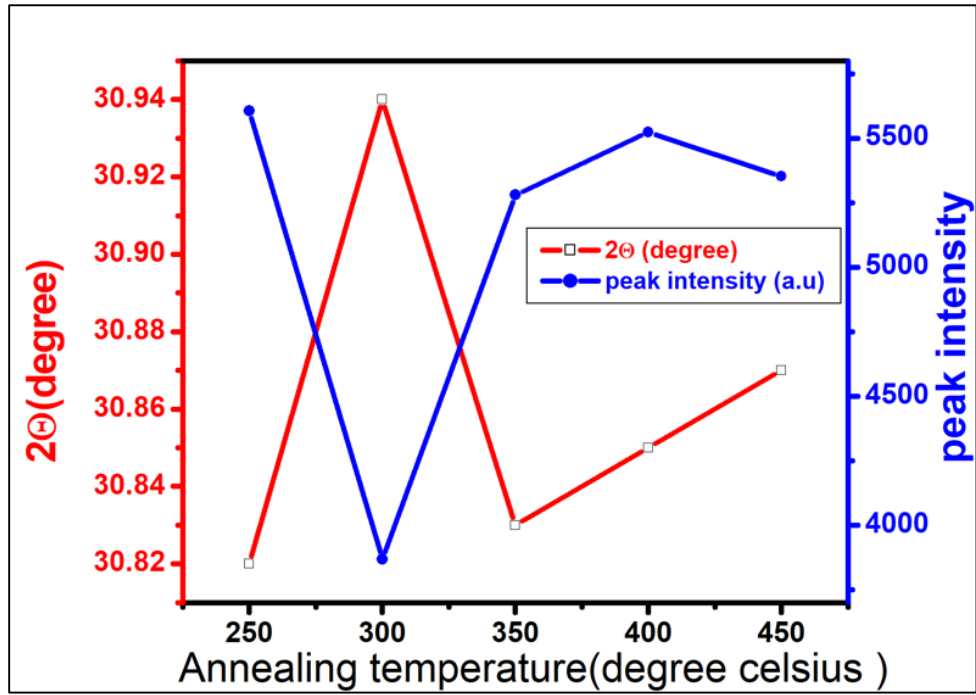


Figure 4.5: Graph of 2 theta and peak intensity of synthesized maghemite nanoparticles at varying calcinating temperature

From the pattern it was observed that calcinating at 250 °C and 300 °C had the highest and lowest intensity respectively. Additionally, peak position of 300 shifted toward a higher angle compared to others. The increase in the diffraction angle with an increase in calcinating temperature confirms changes in the nanoparticle standard lattice parameters of maghemite brought by imperfections and defects within the material. The shift to highest diffraction angle were attributed to decrease in the crystallite sizes caused by decrease in the lattice parameters. The crystallite size was calculated using Debye Scherrer equation (Equation 4.5) (Kiprotich *et al.*, 2025).

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (\text{Equation 4.5})$$

Where λ is the incident wavelength (1.5406 Å), β is the full width at half maximum (rads), θ is the Bragg diffraction angle, D is the crystallite size (nm), and k is the Scherer constant (0.9). Table 4.6 shows nano-sized particles with average diameters; 15.91, 15.12, 12.80, 14.77 and 13.80 nm for 250, 300, 350, 400 and 450 °C calcinating temperature respectively.

Table 4.6: FWHM, crystallite size D (nm), 2 theta value and average D value (nm)

Calcination Temperature (°C)	250	300	350	400	450
2θ (220)	30.82	30.94	30.83	30.85	30.87
FWHM	0.4586	1.55987	0.50589	0.4180	0.44742
D (nm)	17.97	14.72	16.29	19.71	18.42
2θ (400)	44.62	44.72	44.44	44.47	44.52
FWHM	0.59347	0.84399	0.55893	0.52562	0.6248
D (nm)	14.46	10.18	15.35	16.33	13.74
2θ (511)	55.41	55.86	55.62	55.68	55.52
FWHM	0.58595	0.62235	1.33045	1.08439	0.56521
D (nm)	15.31	14.45	6.75	8.28	9.51
D (average)(nm)	15.91	13.12	12.80	14.77	13.89

Similar results were reported by Sum *et al* (2004). The sample annealed at 350 °C registered a smallest crystallite size of 12.80 nm while sample annealed at 250 showed the largest crystallite size of 15.91 nm. The decrease in crystallite size with increasing annealing temperature can be associated with enhanced crystallinity, reduction of lattice imperfections, and reorganization of crystal structures during heat treatment. Moderate annealing promoted atomic mobility and better structural alignment, resulting in the development of smaller and more homogeneous crystallites (Tanwar *et al.*, 2026).

The crystallite size of nanoparticles is affected by the size of the FWHM of the XRD peaks. Figure 4.6 shows the relationship between the crystallite size (nm) and FWHM of synthesized maghemite nanoparticles at varied annealing temperature. The full half at half maximum is inversely proportional to the crystallite size causing a large crystallite size at minimum FWHM. Similar observation was observed by Kiprotich *et al.*, (2025).

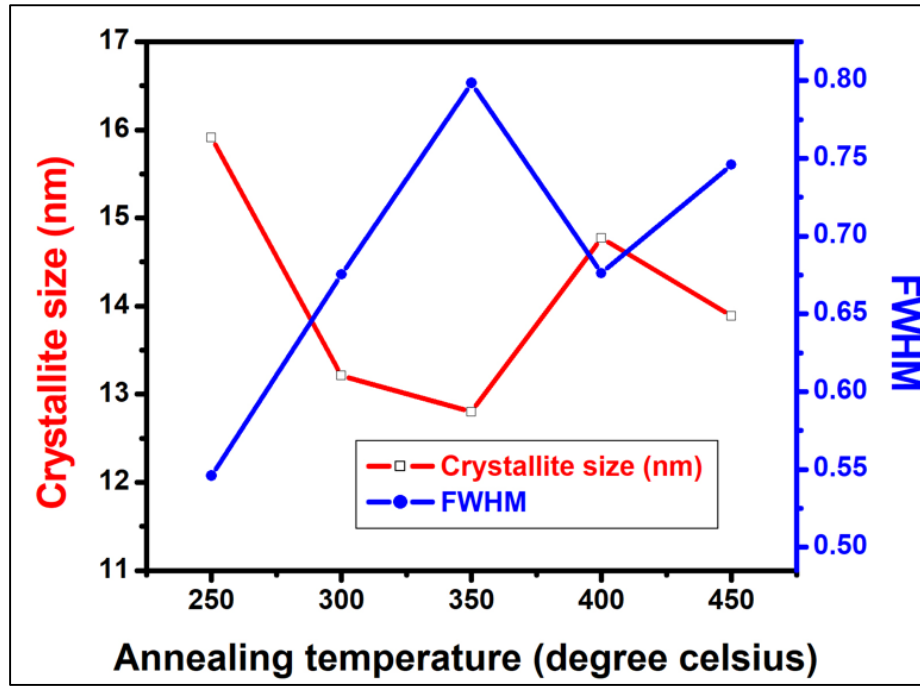


Figure 4.6: Crystallite size (nm) and FWHM at varying calcinating temperature

From Figure 4.6, it is observed that annealing temperature cause a decrease in crystallite size and increases the FWHM. When crystallite is smaller, the number of unit cells contributing to the diffraction peak is reduced leading to broader peaks and higher FWHM. Additionally, the inverse relationship is due to smaller crystallites having a higher proportion of surface atoms and fewer internal planes, leading to increased disorder within the crystal structure. As this disorder changes, it results in variations in the position of the atoms, causing broader peak. The dislocation density (δ), defined as the length of dislocation-lines per unit volume of the crystal was calculated using Equation 4.6 and the results are shown in Tables 4.7 (Bett & Kiprotich, 2024).

Table 4.7: Calculated crystallite size D (nm), FWHM, dislocation density (δ) and strain

Calcination temperature ($^{\circ}\text{C}$)	250	300	350	400	450
FWHM (rads)	0.5460	0.6754	0.7984	0.6760	0.74581
D (nm)	15.91	13.21	12.80	14.77	13.89
δ	0.0039	0.0058	0.0061	0.0046	0.0052
Strain	0.2288	0.2817	0.3344	0.2829	0.3119

The dislocation network within the structure is indicated by the dislocation density and is inversely proportional to crystallite size. To estimate the lattice strain, Williamson and Hall equation was used. The XRD pattern of the prepared nanoparticles deviated from the standard and this can be associated with the stress and strain. The strain was calculated using equation 4.7 (Bett & Kiprotich, 2024).

$$\delta = \frac{1}{D^2} \quad (\text{Equation 4.6})$$

$$\varepsilon = \frac{\beta}{4 \tan \theta} \quad (\text{Equation 4.7})$$

Where ε is the strain and β is the FWHM and θ is the peak position, δ is the dislocation density and D is the crystallite size. Figure 4.7 shows dislocation density and strain effects on the nano-crystal structure. From Figure 4.7, it is observed that micro strain is directly proportional to dislocation.

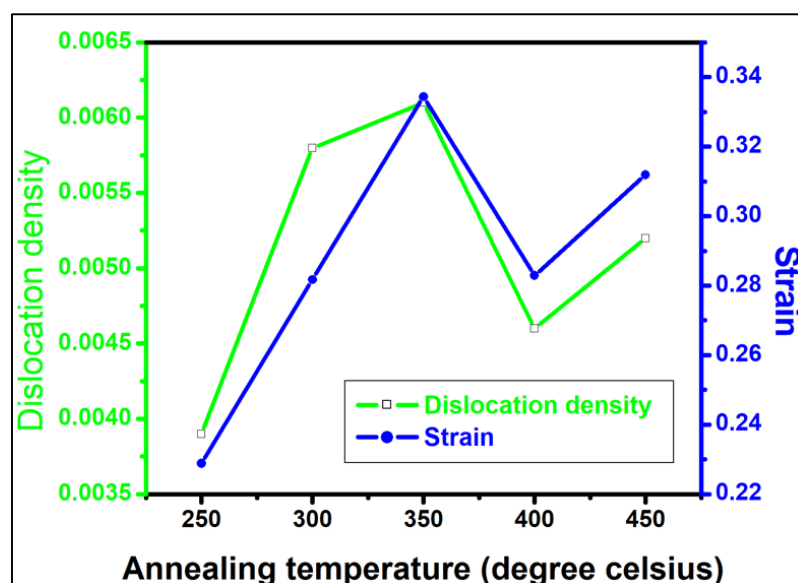


Figure 4.7: Figure shows graph of crystallite size (nm) and FWHM at varying calcinating temperature

Figure 4.8 shows the variation of crystallite size, strain and variation in calcinating temperature. From the figure it is observed that crystallite size is inversely proportional to strain ($D \propto \frac{1}{\epsilon}$).

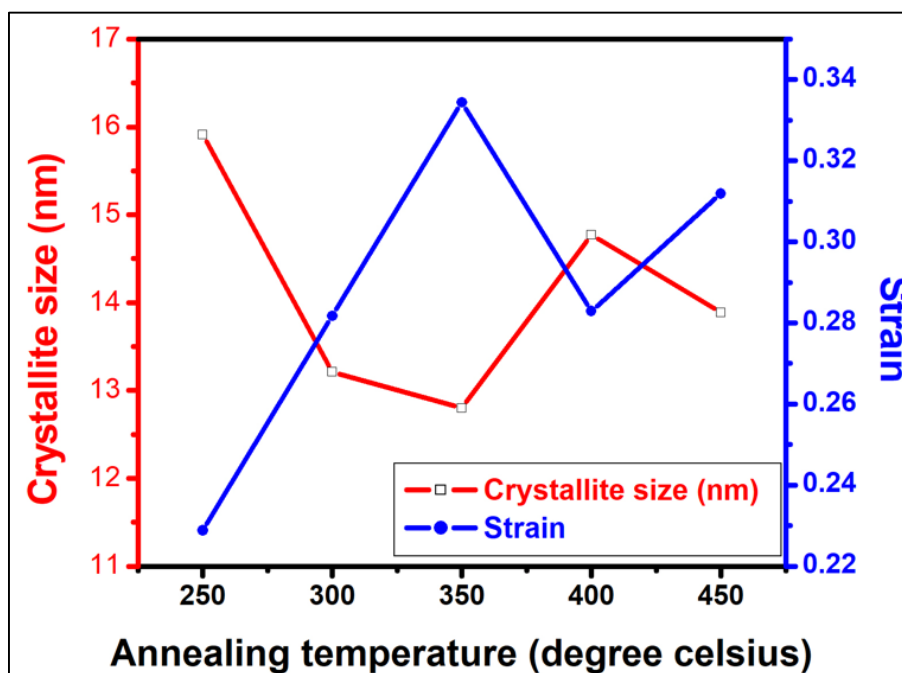


Figure 4.8: Crystallite size (nm) and strain at varying calcinating temperature

From the XRD results, the sample calcinated at 350 °C exhibited the smallest crystallite size together with the highest full width at half maximum (FWHM) and lattice strain, whereas the sample calcinated at 300 °C showed the largest crystallite size accompanied by the lowest FWHM and strain values. This behavior can be explained by the inverse relationship between crystallite size and peak broadening in X-ray Diffraction analysis. At 350 °C, increased thermal energy may have enhanced structural rearrangement, defect redistribution, and microstrain within the crystal lattice, leading to broader diffraction peaks and consequently smaller calculated crystallite sizes according to the Debye Scherrer Equation 4.5.

The higher FWHM observed at 350 °C indicates greater peak broadening, which is commonly associated with reduced crystallite dimensions and increased lattice distortion. In contrast, the sample annealed at 300 °C experienced improved crystal growth and lower lattice imperfections, resulting in sharper diffraction peaks, lower strain, and larger crystallite size. The reduced FWHM at 300 °C suggests enhanced crystallinity and better structural ordering of the maghemite nanoparticles. Therefore, moderate calcination at 300 °C favored crystal growth and stabilization, while further increase to 350 °C introduced structural distortion and refinement of crystallite domains.

4.3.3 Characterization of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles using FTIR

The functional groups present in the green synthesized using pineapple (*Ananas comosus*) peels extract capped and uncapped maghemite was analyzed with Fourier transform infrared spectroscopy (Shimadzu model) under ambient conditions at a range of 400-4000 cm^{-1} in transmittance mode. Fourier transform spectrometer analyzed the functional characteristics of the maghemite nanoparticles calcined at a temperature of 250, 300, 350, 400 and 450 °C. Figure 4.9 shows the FTIR spectra of maghemite $\gamma\text{Fe}_2\text{O}_3$ nanoparticle that was synthesized without the pineapple peel extract and calcined at a temperature of 350 °C.

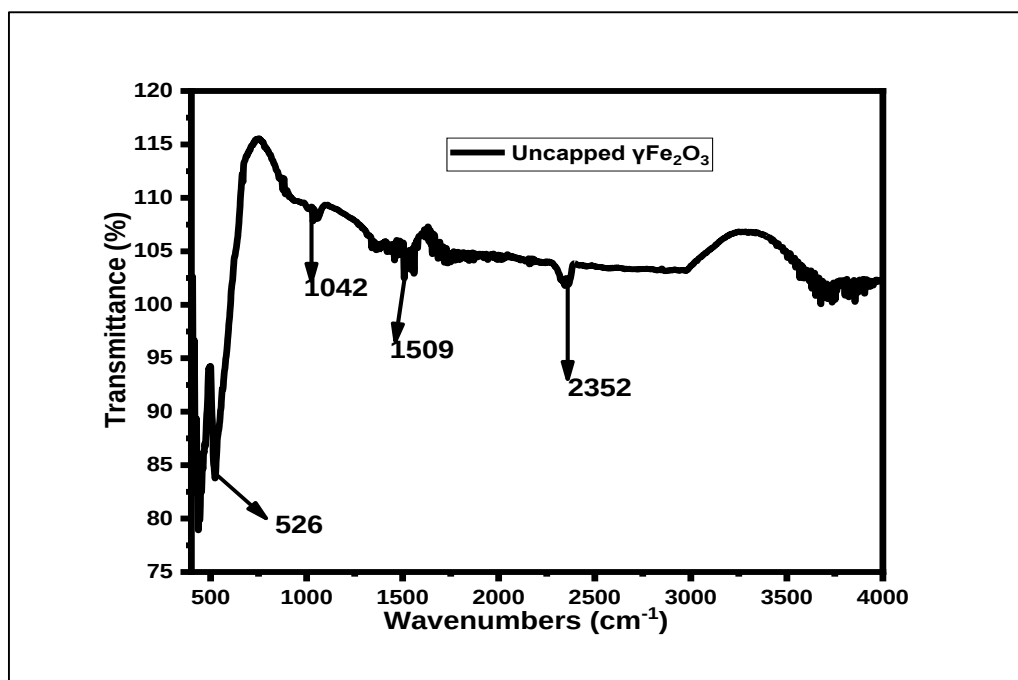


Figure 4.9: The FTIR spectra of uncapped $\gamma\text{Fe}_2\text{O}_3$ nanoparticle calcined at 350 °C

The presence of maghemite nanoparticles can be confirmed by the presence of two strong absorption bands at 426 and 526 cm^{-1} assigned to Fe-O functional groups. The weak vibration band at 1042 and 1509 cm^{-1} are attributed by symmetric and asymmetric vibration of C-O of carboxylate groups that survived calcination at the temperature of 350 °C. The small peak at 2352 cm^{-1} is associated with the asymmetric stretching vibration of atmospheric CO_2 that was present in the atmosphere during analysis. Similar results had been reported by Stoia *et al.*, (2016) when thermal decomposition method was used to prepare $\gamma\text{Fe}_2\text{O}_3$ nanoparticles, the stretching vibration of Fe-O bond was observed at 586 cm^{-1} . When the synthesized $\gamma\text{Fe}_2\text{O}_3$ nanoparticle were analyzed by FTIR prior to calcination, the resulting spectra is presented in Figure 4.10. A broad absorption band centred around 3285 cm^{-1} assigned to O-H stretching vibration was observed (Nnadozie & Ajibade, 2022).

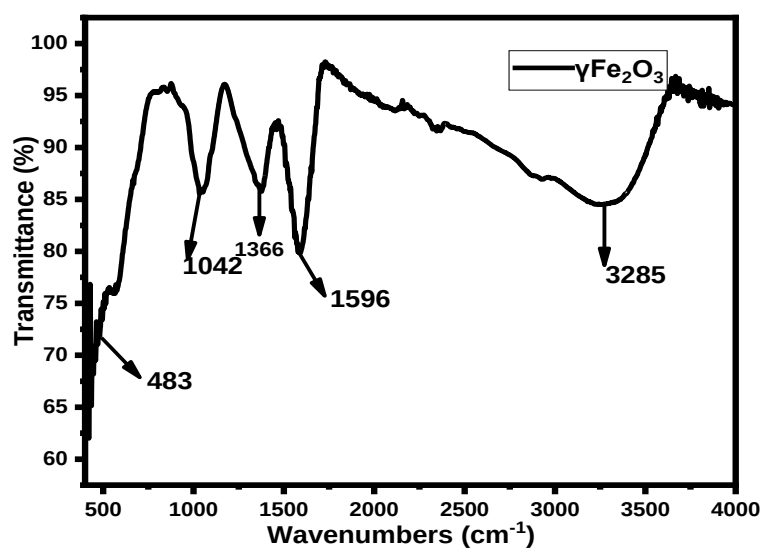


Figure 4.10: The FTIR spectra of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles that was not calcinated.

The band confirms the presence of surface hydroxyl group physically adsorbed water on the surface of nanoparticles. The bending vibration of H-O-H of adsorbed water molecules, typically belonging to nanoparticles due to their high surface area to affinity for moisture were observed at 1596 cm^{-1} . The prominent band at 583 cm^{-1} is attributed to Fe-O stretching vibration, this band confirms the successful formation of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles (Senthilkumar *et al.*, 2021).

The absorption peak at 1042 and 1366 cm^{-1} are associated with stretching and deformation vibration of CO and O-H. The peaks obtained were sharp and broad as compared to $\gamma\text{Fe}_2\text{O}_3$ that was calcinated. When nanoparticles were annealed, the intensity of the peaks decreased and this is attributed to thermal decomposition of phytochemical at high temperature (Sampath & Charles, 2024). This suggest that phytochemicals from plant extract, initially responsible for stabilization of maghemite nanoparticles were decomposed at elevated temperature leaving more crystalline iron oxide phases (Roy *et al.*, 2021).

The influence of calcination temperature on maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles was investigated with FTIR spectroscopy to evaluate the structural impacts of calcination at 250, 300, 350, 400 and 450 °C. The spectra obtained are shown in Figure 4.11.

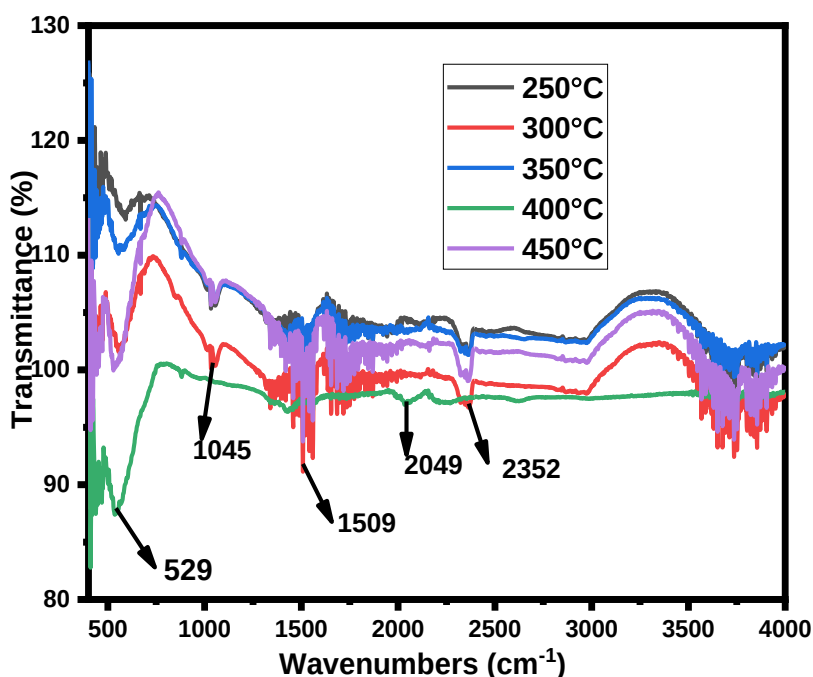


Figure 4.11: The FTIR of $\gamma\text{Fe}_2\text{O}_3$ nanoparticles calcinated at different temperatures

The band observed 529 cm^{-1} correspond to Fe-O stretching bond, characteristics of the iron oxide lattice (Nnadozie & Ajibade, 2022). At high calcination temperature of 400-450 °C, Fe-O band is more defined and slightly sharper, this indicates improved crystallinity and enhanced ordering within the maghemite structure. At lower calcination temperature 200-300 °C a more pronounced peak is observed at 1045 cm^{-1} assigned to CO stretching vibration, as the temperature increased the intensity of the peak decrease which suggest thermal decomposition and removal of organic remnants occurred. At 1509 cm^{-1} a band assigned to O-H bending vibration was observed and its intensity decreased with an increase in calcination temperature which reflects the loss of surface hydroxyl groups. The additional peaks observed at 2352 cm^{-1} in samples calcined at 250, 300, 350 and 400 °C and at 2049 cm^{-1} observed in sample calcined at

400 °C is assigned to adsorbed CO₂. These bands tend to diminish at high temperature when material become thermally stable (Sampath & Charles, 2024).

In the higher wavenumber region 3000-3500 cm⁻¹ a broad O-H stretching band was observed in all samples except in samples calcinated at 400 °C. The peak is more prominent at lower calcination temperature due to hydroxyl groups. At 400 °C the organic compound present in the capping agents have all evaporated and the spectra obtained had low intensity and more pronounced Fe-O stretching band. The decreased in the vibration intensity at high temperature is associated with thermal decomposition that takes place at high calcination temperature that remove physically adsorbed water and organic capping agents (Tran *et al.*, 2022). Fouda *et al.*, (2021) used the *Penicillium expansum* to prepare γ Fe₂O₃ nanoparticles and the FTIR spectra shows similar result with the functional groups obtained in this data, Fe-O at 517 cm⁻¹ and the hydroxyl group at 3375 cm⁻¹.

4.3.4 Characterization of maghemite(γ Fe₂O₃) nanoparticles using UV-Vis

Evolution One Plus Ultraviolet visible spectroscopy (UV-Vis) model was used to study how calcination temperature affects the optical properties of pineapple capped maghemite nanoparticles. The spectra obtained showed that samples calcinated at 400, 450, 350 °C and uncapped samples calcinated at 350 °C had high absorbance in deep UV-region less than 280 nm as shown in Figure 4.11. This is characteristic of iron oxide nanoparticles due to ligand-to-metal charge transfer (LMTC) and O²⁻ → Fe³⁺ (Xu *et al.*, 2022).

Uncapped maghemite (γ Fe₂O₃) nanoparticles had the highest overall absorbance and showed two prominent peaks at 275 and 374 nm. The uncapped and uncalcined samples show significantly higher absorbance, indicating a high concentration of surface bound organic moieties that modified the surface and influence the electronic transition of maghemite nanoparticles (Niraimathee *et al.*, 2016). As the calcination temperature increase from 250-450 °C, a progressive decrease in absorbance was observed.

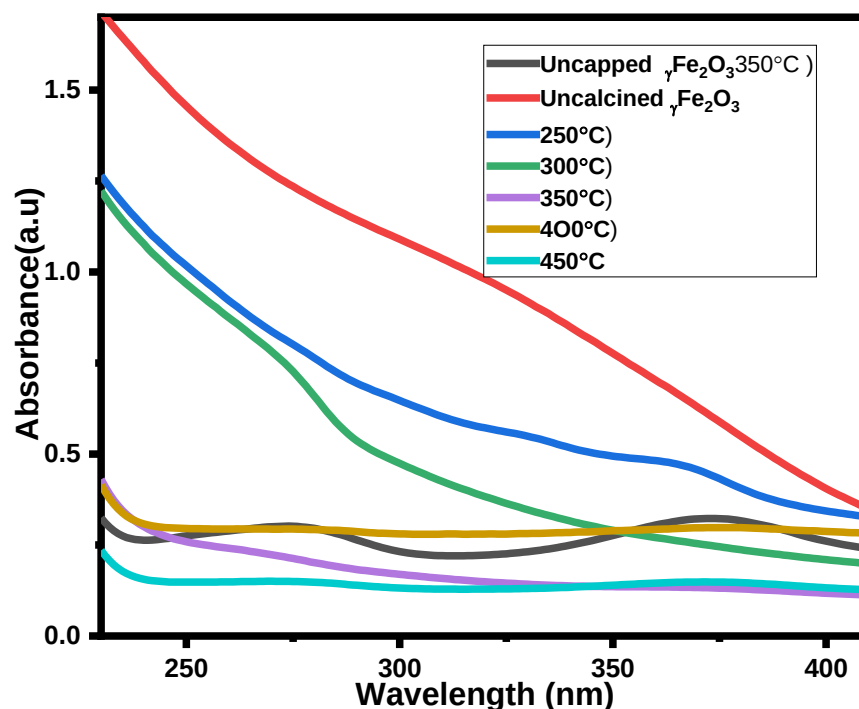


Figure 4.12: The UV-Vis spectra of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles calcinated at different temperatures.

The surface bound biomolecules facilitate the removal of aluminium ions through chelation on complex mechanism, while the phenolic contaminants are removed via π - π interaction and hydrogen bonding (Camargos *et al.*, 2025). As calcination temperature increases, the progressive loss of organic capping agents reduces these interactions, shifting the dominant removal mechanism towards adsorption onto Fe-O surface site (Getahun *et al.*, 2022).

The decrease in absorbance with increase in temperature is associated with the fact that at high calcination temperature, the organic capping molecules are decomposed, therefore, there is reduced surface-bound phytochemicals thus an increase in crystallinity and phase purity of the nanoparticles (Oves *et al.*, 2020). At 400 and 450 °C the samples showed a relatively flat, low absorbance curves, this suggests that the removal of most organic residues and formation of more crystalline $\gamma\text{Fe}_2\text{O}_3$ nanoparticles with fewer surface defects had been achieved (Parsianpour *et al.*, 2015).

Similar result was also observed in FTIR when the calcination temperature was high the peak intensity of the bands decreased, while the un-calcinated samples exhibited a greater number of functional groups due to the presence of surface-bound organic compounds. Calcination helps in decomposing organic molecules from the peel extract and thus improving the crystallinity of nanoparticles (Roy *et al.*, 2021; Sampath & Charles, 2024).

4.4 Adsorption of residual aluminium ion using maghemite nanoparticles

Adsorption residual aluminium ions from water samples were carried out at varying physical conditions. Optimization was done by varying pH, Temperature and contact time as described in section 3.4.

4.4.1 Effect of change in pH on adsorption of residual aluminium ions using maghemite

Residual aluminium ions present in treated water can be remediated by various methods such as Ion exchange, electrochemical degradation, photo-fenton oxidation, reverse osmosis among others (Murage *et al.*, 2022; Oladoye, 2022). In this study, maghemite nanoparticles ($\gamma\text{Fe}_2\text{O}_3$) was used as adsorbent to remove the metal ions from water. For this study de-ionized water had been treated using the homogenous coagulant mixture. Homogenous coagulant mixture had been prepared by mixing all the four coagulants from different manufacturers at ratio of 1:1. During adsorption of residual aluminium 0.5 g of maghemite nanoparticles were used to adsorb residual metal ions from 25 cm³ of the treated water samples.

The pH of the solution was varied while other physical conditions such as the adsorbent dosage, adsorption temperature of 25 °C and contact time of 30 minutes were maintained constant. After batch adsorption was done, percentage adsorption was calculated and results observed are as shown by Table 4.8.

Table 4.8: Effect of pH on percentage adsorption of residual aluminum removal

Parameter	pH	Mean Residual Conc. (ppm)	Mean % Adsorption	p-value (t-test)
Homogeneous Coagulant Mixture	pH 4	6.043±0.18 ^a	56.68±1.70 ^a	0.028
	pH 5	6.624±0.20 ^a	52.51±1.58 ^a	0.035
	pH 6	6.373±0.19 ^a	54.31±1.63 ^a	0.041
	pH 7	8.074±0.24 ^b	42.12±1.26 ^b	<0.001
	pH 8	9.457±0.28 ^c	32.20±0.97 ^c	<0.001
	pH 9	9.390±0.28 ^c	32.68±0.98 ^c	<0.001
p-value (ANOVA)		<0.001	<0.001	

In the Table 4.8, the superscript letters (^{a, b, c}) indicate significant differences between groups ($p < 0.05$). the superscript marked “^a” indicates similar performance, for example pH 4-6 exhibits similar adsorption. Different superscripts indicated different adsorption performance, for example in pH 7 and 9. The ANOVA p-value < 0.001 indicates highly significant pH effect.

The study reported that at low pH of 4, the percentage adsorption was highest at 56.68 %, at pH of 6 and 8 gave 54.31 and 32.20 % respectively. The results showed a decrease in percentage adsorption with increase in pH of the solution. This was explained by formation of aluminium hydroxide with increase in pH reducing percentage adsorption. In addition, formation of electrostatic bonds between maghemite surface with hydroxide ions also reduces the number of active adsorption sites available (Hou *et al.*, 2020; Molinari *et al.*, 2020). This can also be demonstrated by line graph of percentage adsorption against pH as shown in Figure 4.13.

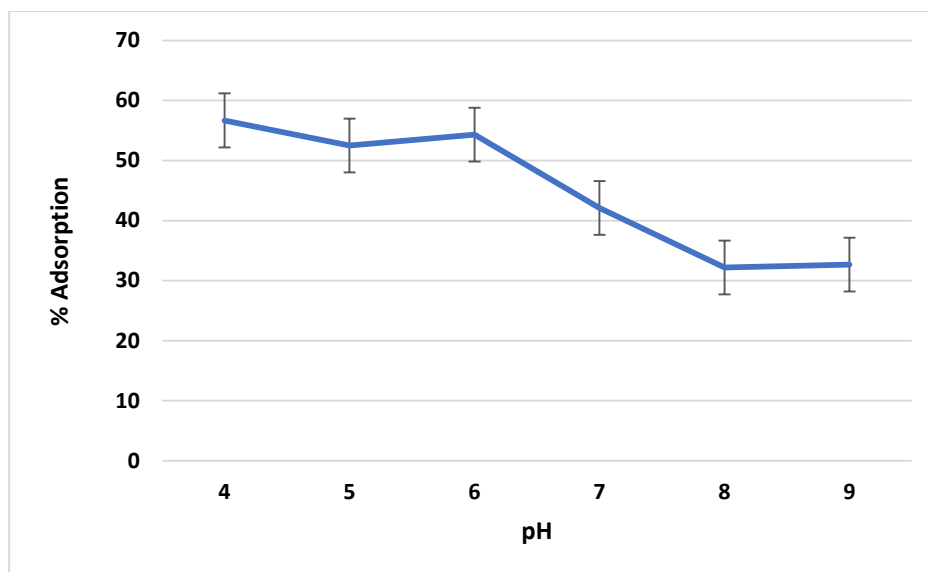


Figure 4.13: Effect of variation of pH on percentage adsorption

Statistical analysis of variance (ANOVA) was carried out to determine statistical relationship between change in pH and percentage adsorption. The results gave a P-value less than 0.001 indicates highly significant pH effect on percentage adsorption (Table 4.6). This concludes that change in pH does statistically affect percentage adsorption at confidence level of 95%. Therefore, there was a strong relationship between variation in pH and rate of adsorption of aluminium ions from treated water using maghemite nanoparticles.

Saddiqa *et al.* (2025), carried out a study on adsorption of Mn^{+2} from water using magnetite-maghemite nanoparticles, pH was found to have a big impact on the rate of adsorption. It was reported that optimum pH for the adsorption was be 6.5. It was reported that at lower pH metal ions experienced competition for adsorption site with H_3O^{+1} ions reducing rate of adsorption. In addition, at very low pH (around 2 or lower) there was high electrostatic repulsion between positive metal ion and positive surface of the adsorbent. However, at pH higher than 7, Mn^{+2} ions were precipitated in form of insoluble hydroxides (Siddiqa *et al.*, 2025).

In a study carried out by Behera *et al.* (2020) on adsorption of arsenic and chromium from water using magnetite-maghemite mixture, they found that adsorption depended on the pH of the solution. They reported that arsenic was best adsorbed at pH of 2. The best adsorption occurred within range of 2 up to 5. Then percentage adsorption decreased gradually with increase in pH and remained constant from 10 which was

the lowest rate of adsorption. These results indicated that adsorption of arsenic(iii) and arsenic(v) occurred best at acidic conditions. These observations are attributed to different species of arsenic formed at different pH with the best adsorbed species occurring at low pH (Behera *et al.*, 2020).

In a study on remediation of water by Beksissa *et al.*, (2021) on adsorption of heavy metals from water, they found that rate of adsorption depends different parameters such as pH and adsorbent dosage. They reported that adsorption of Cr^{+6} ions decreased with increase in pH. While adsorption of Cu^{+2} and Ni^{+2} decreased with increase in pH. It was reported that Cr^{+6} ions were adsorbed best at pH of 3. This was attributed to the fact that Cr^{+6} exists in form of CrO_4^{-2} in solution. As pH increases there is gradual increase in concentration of OH^- which competes with CrO_4^{-2} ions for the adsorption sites on the maghemite nanoparticles. However, for copper and nickel ions the adsorption increases with increase in pH. Copper is adsorbed best at pH of 7, and 10 for nickel. This observation is attributed to decrease in concentration of H^{+1} ions which competes for adsorption sites with the metal ions since adsorption surface is negatively charged (Tiadi *et al.*, 2020; Beksissa *et al.*, 2021).

A study on adsorption of arsenic from natural waters using maghemite nanoparticles, it was found an efficient method of water remediation. The study reported that the rate of adsorption depended on pH. In return the pH is determined by speciation of the metal and point of zero charge (pH_{pzc}). Point of zero charge is the pH at which the net electrical charge of surface is zero. In a solution, at lower pH, the more positive the surface of the adsorbent (Kumar *et al.*, 2022; Sufiani *et al.*, 2023). This increased the rate of adsorption of arsenic which is in form of $\text{H}_2\text{AsO}_4^{-1}$ at low pH. The study found that increase in pH, resulted in decrease in rate of adsorption, this was attributed to pH value exceeding pH_{pzc} value. The adsorbents surface becomes negative which repels $\text{H}_2\text{AsO}_4^{-1}$ reducing adsorption (Kumar *et al.*, 2022).

4.4.2 Effect of change in temperature on adsorption of residual aluminium ions

Batch adsorption was carried at various temperature range. This study was at same conditions of pH of 5 and contact time of 30 minutes. On addition of 0.5 g of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles adsorbent the mixture was stirred at constant speed throughout the study. On analysis of the concentration of residual aluminium ions after

adsorption, following data was obtained as illustrated in the Table 4.9. Initial concentration of aluminium ions before adsorption was 13.9486 ppm.

Table 4.9: Effects of variation of temperature on percentage adsorption.

Parameter	Temperature (°C)	Mean Residual Conc. (ppm)	Mean % Adsorption	p-value (t-test)
Homogeneous Coagulant Mixture	35 °C	6.558±0.20 ^a	52.99±1.59 ^a	0.156
	45 °C	6.799±0.20 ^a	51.26±1.54 ^a	0.148
	55 °C	8.703±0.26 ^b	36.60±1.10 ^b	<0.001
	65 °C	6.049±0.18 ^a	56.64±1.70 ^c	<0.001
	75 °C	7.724±0.23 ^{ab}	44.63±1.34 ^{ab}	0.012
p-value (ANOVA)		<0.001	<0.001	

In the Table 4.9, the superscript ^a (35, 45, 65 °C) indicates similar low residual concentration. Superscript ^b (55 °C) shows significantly higher residual. Best adsorption at 65 °C (superscript ^c). Intermediate superscript ^{ab} indicates transitional values.

Statistical analysis of data obtained on effect of variation of temperature was carried out. Correlation between variation in temperature and percentage adsorption was determined. The results demonstrated a slight decrease in rate of adsorption from 35 °C to 55 °C. However, the adsorption increased from 55 to 65 °C. This was followed by a decrease confirming erratic behavior of effect of temperature change on the rate of adsorption. The peak performance was reported at 65 °C (56.64 %). The is illustrated by the graph of change in percentage adsorption against temperature (°C) is as shown in Figure 4.14.

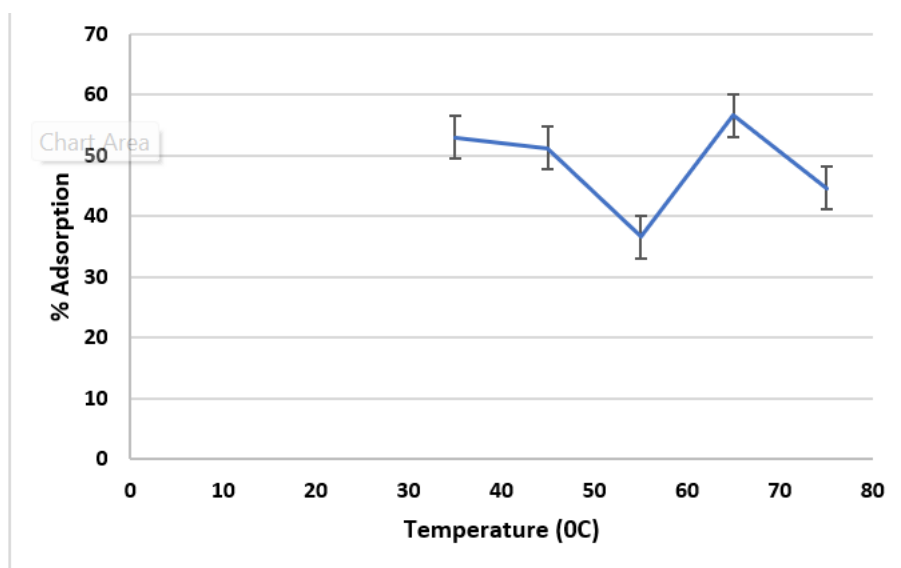


Figure 4.14: Effect of variation of temperature on percentage adsorption

From the graph in Figure 4.14, it demonstrated a weak negative correlation between temperature and percentage adsorption. Using sample mean percentage adsorption, and sum of squares, Pearson's correlation coefficient was determined. The data gave correlation coefficient (r), which was found to be -0.3 . These results indicated a weak correlation between adsorption temperature and percentage adsorption. The results exhibited moderate sensitivity to temperature change. The data analysis concluded that there is a slight tendency for percentage adsorption to decrease with increase in adsorption temperature. However, this relationship is not strong or consistent. This complex pattern suggests competing thermodynamic factors: increasing temperature enhances reaction kinetics but may also increase aluminum complex solubility and reduce coagulant stability beyond optimal temperature.

Similar observations were made by Siddiqua *et al.*, (2025) in adsorption of Mn^{+2} ions from water using magnetite-maghemite nanoparticles. The researchers reported that adsorption of Mn^{+2} was endothermic at low temperatures. This resulted in stronger metal ions-adsorbent binding explaining relatively higher rate of adsorption. However, as the temperature increased, there was general decrease in the rate of adsorption due to weak binding as a result of high kinetic energy of the metal ions in solution. It was observed that the effect of temperature change exhibited some randomness especially at higher temperature. This was attributed to disorderliness of molecules

during formation of adsorbate-adsorbent complex as well as involvement of water in the binding process (Siddiqa *et al.*, 2025).

In a study in competitive removal of Cu, Ni, Mn(ii), and Cr(vi) ions from acid mine drainage using maghemite, different adsorption physical conditions were applied. Percentage adsorption was carried out within temperature range of 20-70 °C. The study reported that there was a slight increase in adsorption from 20 to 30 °C. However, adsorption remained almost constant up to 70 °C. There it was concluded that change in temperature had just slight improvement in adsorption of all the five metal ions studied, while change in pH had the largest effect (Etale *et al.*, 2016).

Previous studies have not reported much on the effect of change in temperature on the adsorptions of metal ions using maghemite nanoparticles from brewed tea waste waters. However, a study on adsorption of lead, zinc Nickel and cadmium from water it was found temperature plays a minor role in percentage adsorption of metal ion. It was reported that optimum adsorption temperatures of all three metal ions to be 45 °C (Çelebi *et al.*, 2020).

4.4.3 Effect of change in contact time on adsorption of residual aluminium ions

Treated water sample analyzed had initial concentration of residual aluminium ion of 13.9486 ppm before adsorption. The batch adsorption was carried out at variable contact time. Adsorption was done by adding 0.5 g of maghemite nanoparticles into analyte water sample and shaken at varied contact time in increasing order from 15, 30, 45, 60 and 75 minutes. On determination of residual aluminium ions after adsorption, it was found that the percentage adsorption increased with increase in contact time. This observation indicated that there was very high rate of adsorption at the initial short time of the interaction between adsorbent and adsorbate. This is caused by availability of large number of adsorption sites on the adsorbents surface. As electrostatic bonds continue to form between adsorbent active sites and adsorbate the rate of adsorption reduces with increase in contact time as illustrated by Table 4.10 and Figure 4.15.

Table 4.10: Effect Variation of contact time on percentage adsorption.

Parameter	Contact Time (min)	Mean Residual Conc. (ppm)	Mean % Adsorption	p-value (t-test)
Homogenous Coagulant Mixture	15 min	8.235±0.25a	40.96±1.23a	<0.001
	30 min	6.387±0.19b	54.21±1.63b	<0.001
	45 min	5.672±0.17c	59.33±1.78c	<0.001
	60 min	4.893±0.15d	64.92±1.95d	<0.001
	75 min	4.564±0.14d	67.28±2.02d	0.067
p-value (ANOVA)		<0.001	<0.001	

In the table 4.10 the superscripts ^{a, b, c} indicates the readings are significantly different: Each time interval from 15-45 min is significantly different. Superscript ^d, representing equilibrium with no significant difference: Reading between 60-75 minutes were not significantly different. P value = 0.067), indicating optimal contact time is 60 minutes.

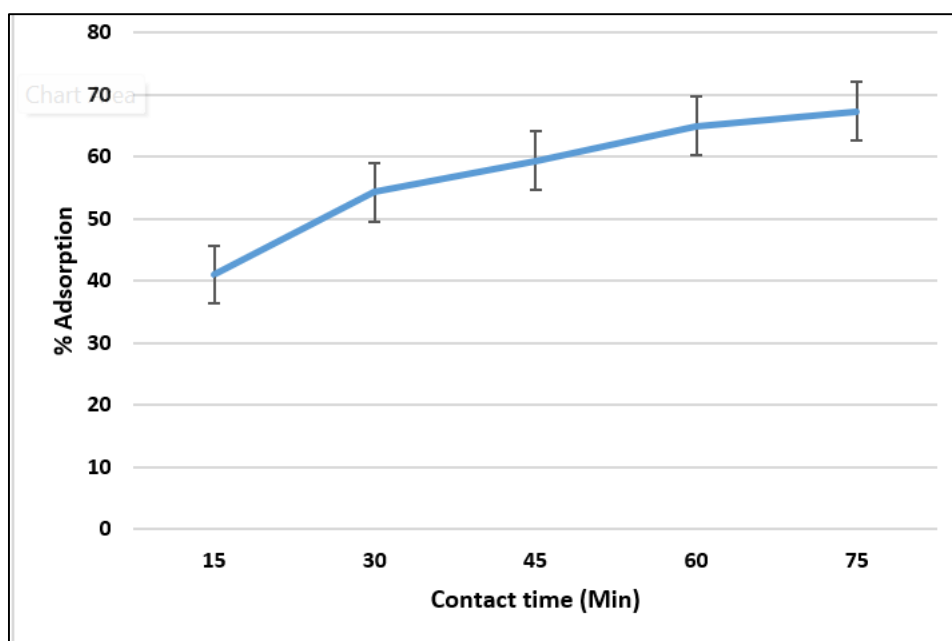


Figure 4.15: Percentage adsorption against contact time

Variation in contact time demonstrates a strong positive correlation with adsorption efficiency. Percentage adsorption increased progressively: 40.96% (15 min) to 54.21% (30 min), to 59.33% (45 min), to 64.92% (60 min) and to 67.28% (75 min). Correspondingly, the residual concentration decreased from 8.23 ppm to 4.56 ppm. The rate of adsorption was highest between 15-30 minutes (13.25% increase) and decreased thereafter, indicating approach toward equilibrium as shown in Figure 4.15. The continuous improvement suggested kinetically controlled adsorption following pseudo-second-order kinetics. This data provided the equilibrium concentrations needed for adsorption isotherm modeling.

In a study on adsorption of Mn^{+2} ion from water using magnetite-maghemite nanoparticles, it was found that contact time had a great effect on the adsorption of the metal ions. When 10 ppm spiked Mn^{+2} solution was used, the researcher reported that highest rate of adsorption occurred at first few minutes. The optimum adsorption occurred at 20 minutes. This observation was attributed to high initial concentration of adsorbate and reduced resistance to mass transfer. Rate of adsorption reduced with increase in contact time due to reduced number of active adsorption sites available (Siddiqa *et al.*, 2025).

Etale *et al.*, (2016) did a study on adsorption of Mn(ii), Cu(ii) and U(vi) ions using maghemite adsorbent, it was reported that rapid adsorption within the first 30 minutes for all three metals. This study was carried out at pH of 3. This was explained by reporting that binding occurred as result of chemisorption and also diffusion of metal ions into adsorption pores on the adsorbents surface (Etale *et al.*, 2016). In a study on adsorption of Cr^{+6} from waste waters using maghemite nanoparticles, it reported that rate of adsorption was highest (90%) at the initial few minutes of contact. This was explained by presence of high initial concentration of the adsorbate. The rate of adsorption decreased with decreased in concentration as adsorption sites reduced as time progressed (Hou *et al.*, 2020).

The researchers reported that metal ions with small particles sizes exhibited higher adsorption rates due to their size than large ions. Cu with atomic radius of 0.419 nm gave the highest rate of adsorption of 56% while U with largest atomic radius of 1.08 nm gave adsorption of 49%. In addition, it was found that adsorption was also affect by the structural configuration of the metal ions (Etale *et al.*, 2016).

4.4.4 Adsorption isotherms models for aluminium ions

The initial concentration of the spiked solution before adsorption was 13.9486 ppm (C_0). After batch adsorption was carried out at variable contact time, residual concentration of aluminium (C_e) was determined using AAS. The amount of aluminium adsorbed (Q_e) was calculated as the difference of “ C_0 ” and “ C_e ”. The equilibrium adsorption data obtained is as shown in Table 4.11.

Table 4.11: Equilibrium adsorption data

Time (min)	Ce (ppm)	Qe (ppm)	Description
15	8.2349	5.7137	Early stage
30	6.3871	7.5615	Mid stage
45	5.6722	8.2764	Approaching equilibrium
60	4.8931	9.0555	Near equilibrium
75	4.5642	9.3844	Equilibrium

In the table above, the symbol “ C_0 ” represents the initial concentration of residual aluminium before adsorption (13.9486 ppm). The symbol “ C_e ” represents the equilibrium concentration(residual) after adsorption. The “ Q_e ” represents the amount of aluminium adsorbed. These quantities are related by Equation 4.8.

$$(Q_e = C_0 - C_e). \quad \text{(Equation 4.8)}$$

In this study adsorption mechanism of aluminum was determined. This was done by evaluating equilibrium data from contact time studies against four standard isotherm models: Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich as shown in Table 4.12.

Table 4.12: Isotherm Model Parameters and Fit Quality

Isotherm Model	Key Parameters	R² Value	Fit Quality
Langmuir	q _{max} = 4.4120 ppm KL = -0.4026 L/ppm	0.910831	Very Good
Freundlich	KF = 32.0733 n = -1.2560	0.974800	Excellent
Temkin	Fit failed	0.000000	Poor
Dubinin-Radushkevich	q _m = 5.0637 ppm K = -2.7×10 ⁻⁶	0.909065	Very Good

In the above table, the symbol “R²” is the correlation coefficient, measuring how well the model fits the experimental data. It ranges from 0 to 1 (or 0% to 100%). R² = 1.00 means perfect fit, R² > 0.95 is excellent, R² > 0.90 is very good. In this study, Freundlich R² = 0.9748 (97.48%) is excellent, meaning the model explains 97.48% of the variation in the adsorption data. The symbol q_{max} = 4.4120 ppm: This is the maximum adsorption capacity. The theoretical limit of how much aluminum can be adsorbed when the surface is completely saturated. The symbol KL = -0.4026 L/ppm: This is the Langmuir constant, which represents the binding affinity or "stickiness" between aluminum and the coagulant surface. The symbol KF = 32.0733: This is the Freundlich constant, representing the adsorption capacity of the material. While symbol n = -1.2560: This is the heterogeneity factor or intensity parameter. It indicates how favorable the adsorption is, - n > 1: Unfavorable adsorption (gets harder as concentration increases). If - n = 1: Linear adsorption (stays the same). While, - 0 < n < 1: Favorable adsorption (gets easier at higher concentrations). If - Negative n: VERY favorable adsorption.

Using key parameters in determination of adsorption isotherms of aluminium ions using maghemite nanoparticles was carried out. In Langmuir isotherm assumes monolayer adsorption on homogeneous surface that is, it assumed that all binding sites of the adsorbent are identical. The good fit suggests some surface uniformity, but negative KL value indicates model limitations for this system (Azizian *et al.*, 2018). In this study, the KL gave a negative value. This indicated the surface of the adsorbent was not homogeneous and likely has different types of binding sites with different

energies. Therefore, the adsorption did not fit into Langmuir adsorption isotherm (Ramos-Guivar *et al.*, 2020).

Freundlich adsorption isotherm is used to describes multilayer adsorption on heterogeneous surfaces. The excellent fit ($R^2 > 0.97$) indicates aluminum adsorption occurs on heterogeneous adsorbent surfaces with varying binding energies. $KF = 32.07$ reflects high adsorption capacity, while $n = -1.26$ indicates favorable adsorption. The surface of the adsorbent has adsorption sites some with strong, medium and others weak binding energies. The strongest binding sites fill first, then progressively weaker sites (Vigdorowitsch *et al.*, 2021).

In this study, a high Freundlich constant ($KF=32.0733$) was obtained indicating better overall adsorption capacity maghemite nanoparticles in removal of aluminium ions from water. Therefore, maghemite is an effective adsorbent in water remediation. This is the heterogeneity factor or intensity parameter which indicates how favorable adsorption. In this study heterogeneity factor had a negative value ($n=-1,2560$) indicating adsorption was very favorable (Vigdorowitsch *et al.*, 2021). Therefore, aluminum ions strongly prefer to bind to the maghemite nanoparticles, and the heterogeneous surface provides multiple high-energy binding sites.

The correlation coefficient (R^2) obtained in this study was highest in Freundlich ($R^2 = 0.9748$). Therefore, the experimental data was a perfect match for Freundlich adsorption isotherm when compared with all other models tested. This indicated aluminium ions bonded on to the adsorbent in multiple layers (Ramos-Guivar *et al.*, 2020). This resulted in 65-67% adsorption efficiency in removal of metal ions from water.

Temkin Isotherm model failed to fit the data, suggesting interaction effects are not the dominant factor ($R^2=0.0000$). The Temkin model assumes that the heat of adsorption decreases linearly as more adsorbate ions are adsorbed due to interactions between adsorbed ions. The complete failure to fit ($R^2 = 0.0000$) indicates that these interaction effects are not the primary mechanism in your aluminum ions removal system. Instead, surface heterogeneity (Freundlich) dominates.

Dubinin-Radushkevich Isotherm model ($R^2 = 0.9091$) gave a very good fit suggests significant contribution from both physical and chemical mechanisms. The $q_m = 5.06$ ppm indicates total aluminum removal potential. The adsorption capacity ($q_m = 5.0637$ ppm) is similar to q_{max} in Langmuir, this represents the maximum adsorption capacity through pore-filling mechanisms. The constant ($K = -2.7 \times 10^{-6}$) is used to calculate the mean free energy of adsorption, which helps determine if the adsorption is physical (weak) or chemical (strong) in nature. The very good fit ($R^2 = 0.9091$) suggests that both physical adsorption (weak van der Waals forces) and chemical adsorption (stronger chemical bonds) contribute to aluminum removal in your system. Comparative graphs of Freundlich and Langmuir isotherms were plotted as shown in Figure 4.16.

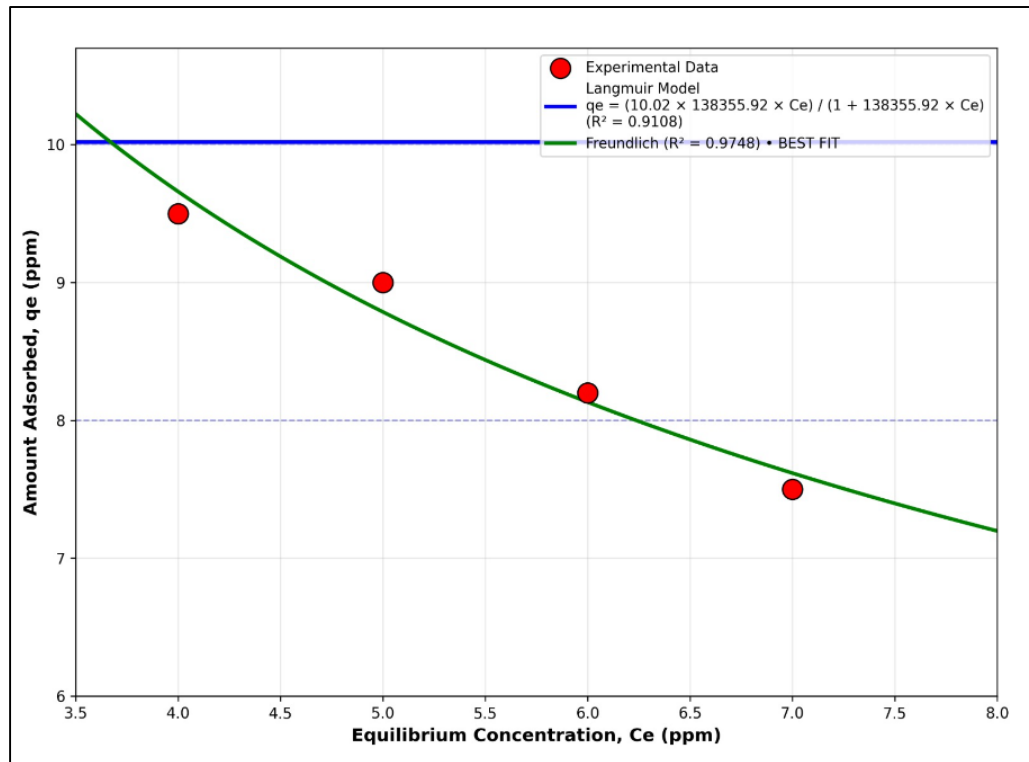


Figure 4.16: Graph comparing Freundlich and Langmuir adsorption isotherms

The Freundlich isotherm provides the best description of aluminum adsorption by Coagulant 2, indicating a heterogeneous adsorption process involving multiple binding sites with different affinities (Chen, 2015; Vigdorowitsch *et al.*, 2021).

4.5 Adsorption of phenolic compounds (phenol and resorcinol) using maghemite nanoparticles

In this study, effectiveness of maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles in remediation of phenolic compounds from water was investigated. Effect of change in pH, temperature and contact time was studied. Spiked solutions of phenol and resorcinol which are some of the most common phenolic compounds and also water pollutants are used in this study.

4.5.1 Validation of UV-Vis

Standard solutions were prepared from stock 100 ppm spiked stock phenol and resorcinol solution as described in section 3.12 above. On analysis of the standard phenol solutions using UV-Vis, table of absorbance against concentrations of standard solutions was obtained as shown in Table 4.13. All readings were done in triplicate (N=3).

Table 4.13: Concentration of standards of phenol and corresponding absorbance

Concentration (ppm)	Absorbance	p-value (t-test)
20	0.3650±0.0109	<0.001
40	0.6760±0.0203	<0.001
60	0.8470±0.0254	<0.001
80	1.2690±0.0381	<0.001
100	1.5520±0.0466	<0.001
p-value (ANOVA): <0.001		

On analysis of the standard phenol solutions using UV-Vis, the table of absorbance against concentrations of standard solutions was obtained as shown in Table 4.14. All readings were done in triplicate (N=3).

Table 4.14: Concentration of standards of resorcinol and corresponding absorbance

Concentration (ppm)	Absorbance	p-value (t-test)
20	0.3770±0.0113	<0.001
30	0.6420±0.0193	<0.001
60	0.9460±0.0284	<0.001
80	1.3420±0.0403	<0.001
100	1.6220±0.0487	<0.001
p-value (ANOVA): <0.001		

The UV-Vis data was used to draw the calibration curve of absorbance against concentration of the standard solutions for phenol and resorcinol are as shown above is as shown in Figure 4.17 and 4.18.

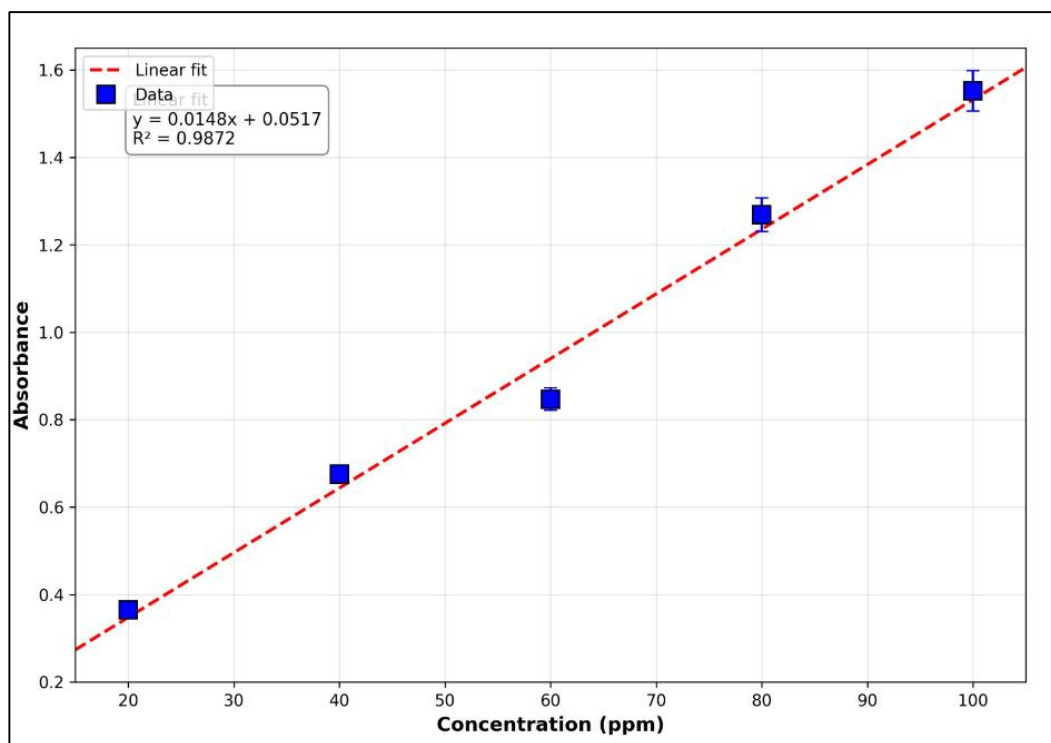


Figure 4.17: Calibration curve of phenol

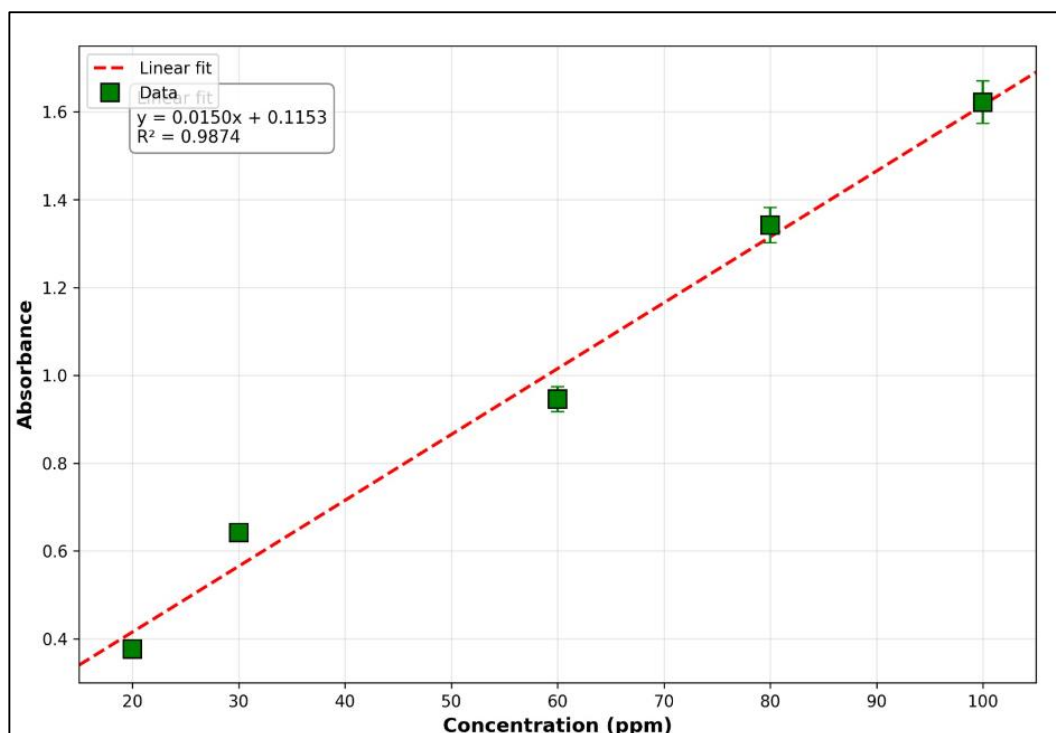


Figure 4.18: Calibration curve of Resorcinol

The calibration curves show excellent linearity for both phenol ($R^2 = 0.9998$) and resorcinol ($R^2 = 1.0000$) with highly significant p-values (<0.001), indicating high precision and reliability. ANOVA confirms significant differences between concentration levels ($p < 0.001$). The linear relationship validates Beer-Lambert law applicability.

4.5.2 Effect of change in pH on adsorption of phenol and resorcinol using maghemite

Batch adsorption of phenol and resorcinol was done by addition of 0.5 g maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles into 25cm^3 of de-ionized water spiked solutions (100 ppm). Adsorption was carried out at different pH ranging from pH 3 to 11. After adsorption and filtration, the filtrate was analyzed using UV-Vis to determine residual concentration of the phenol. On analysis it was found maghemite nanoparticles ($\gamma\text{Fe}_2\text{O}_3$) was an effective adsorbent of phenol and resorcinol molecules from water. The data obtained is as illustrated in Table 4.15 and 4.16.

Table 4.15: Effect variation of pH on percentage adsorption of phenol (Initial concentration is 100 ppm).

pH	Residual conc (ppm)	p-value	%Adsorption	p-value.1
3	42.91±1.72	<0.001	57.09±2.28	<0.001
5	40.87±1.63	<0.001	59.13±2.37	<0.001
7	49.63±1.99	<0.001	50.37±2.01	<0.001
9	65.02±2.60	<0.001	34.98±1.40	<0.001
11	71.45±2.86	<0.001	28.55±1.14	<0.001
p-value (ANOVA): <0.001				

Table 4.16: Effect variation of pH on percentage adsorption of resorcinol (Initial concentration is 100 ppm).

pH	Residual conc (ppm)	p-value	%Adsorption	p-value.1
3	46.27±1.85	<0.001	53.73±2.15	<0.001
5	36.95±1.48	<0.001	63.05±2.52	<0.001
7	31.04±1.24	<0.001	68.96±2.76	<0.001
9	32.94±1.32	<0.001	67.06±2.68	<0.001
11	43.66±1.75	<0.001	56.34±2.25	<0.001
p-value (ANOVA): <0.001				

The study found that at low pH (3), adsorption of phenol was 57.09 %. However, the optimum pH was 5 where percentage adsorption was 59.13%. Then percentage adsorption decreased significantly. At pH of 11, percentage adsorption was lowest at 28.55 %. Adsorption of phenol exhibited a general decline in percentage adsorption from optimum pH of 5. In batch adsorption of resorcinol percentage adsorption at pH 3 was 53.73 %. It increased steadily to a maximum of 68.96 % at pH 7. Then gradually decreased to 56.34 % at pH 11. This trend of effect of change in pH on the percentage adsorption of both phenol and resorcinol is as shown in Table 4.15 and Table 4.16 is illustrated further by the curve of change in pH against adsorption by Figure 4.19.

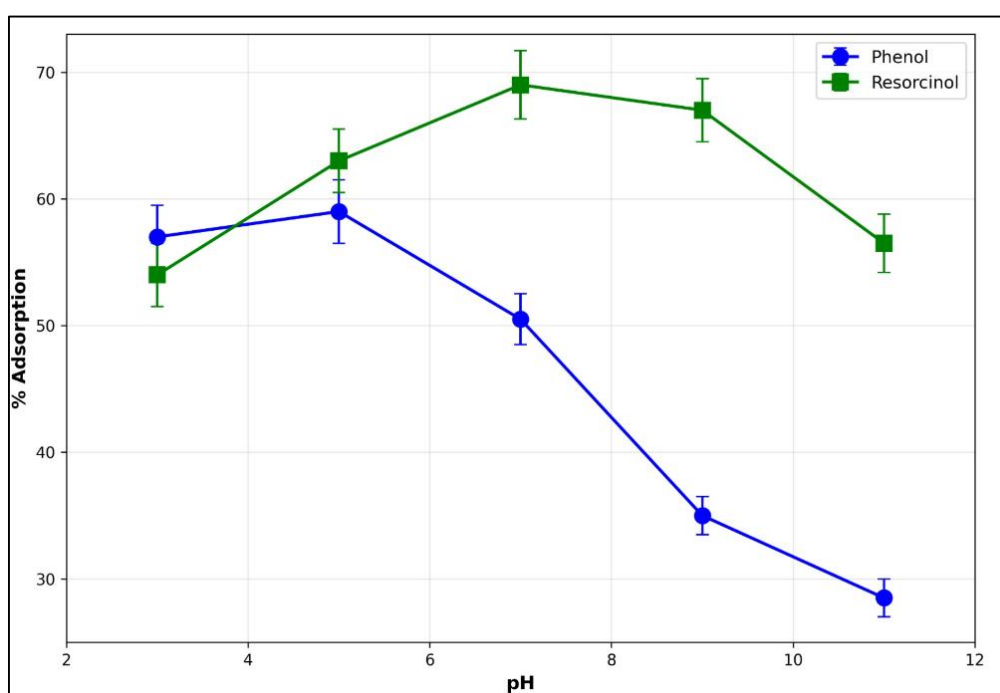


Figure 4.19: Change in percentage adsorption against pH

For both phenol and resorcinol, it was observed that adsorption was low at higher pH greater than 7. This was attributed to anionic repulsion at alkaline environment between hydroxyl group negative adsorbent surface. The maximum percentage adsorption of resorcinol ($68.96 \pm 2.76\%$) at pH 7, maintaining 63-68% between pH 5-9. Statistical significance ($p < 0.001$) validates broader optimal pH range due to dual hydroxyl groups.

The decrease in percentage adsorption with increase in pH was attributed to adsorption kinetics. Point of zero charge (pH_{pzc}) of maghemite is around pH 7. At pH below point of zero charge maghemite nanoparticles will have a positive charge, therefore it forms

H-bonds with hydroxy group of the phenol. These bonds are more pronounced at low pH resulting in high adsorption capacity. Similarly, as pH increases beyond point of zero charge, the maghemite acquires a negative charge reducing ability to form H-bonds. Therefore, rate of adsorption reduced with increase in pH (Dehmani *et al.*, 2020).

In a study on adsorption of phenol from water using superparamagnetic iron oxide (Fe_3O_4) nanoparticles, it was reported that change in pH had a great effect on the rate of adsorption. The optimum pH for adsorption was found to be 3, where percentage adsorption of 95% was achieved at 60 minutes of contact time. These observations were attributed to photo catalytic decomposition of phenol molecules at lower pH to form OH and O_2 radicals in presence of Ultra-Violet (UV) radiations. The rate of decomposition of phenol decreases with increase in pH (Bazrafshan *et al.*, 2023).

In another study on adsorption of phenol from aqueous solution using iron oxide ($\alpha\text{Fe}_2\text{O}_3$) nanoparticles (hematite), change in pH was found to play a major role in rate of adsorption. It reported that adsorption efficiency decreased from pH higher than 7. At a pH below point of zero charge of hematite (pH_{pzc}) which is 7.1, the surface of the adsorbent has a net positive charge. And it has negative at pH above pH_{pzc} the adsorbent has a negative charge on its surface. Therefore, at low pH, there is formation of strong π - π bonds between adsorbent and phenol molecules. This results in high adsorption efficiency at pH values lower than 7. At higher pH environment (above 8), phenol exists as phenolate this results in charge repulsion between the phenolate and hematite surface reducing adsorption efficiency at higher pH. This behavior is very similar to that of maghemite nanoparticles (Dehmani *et al.*, 2020).

4.5.3 Effect of change in contact time on adsorption of phenol and resorcinol using maghemite

Batch adsorption was carried out by using 0.5 g of maghemite nanoparticles adsorbent on 25 cm³ of 100 ppm phenol and resorcinol stock solutions separately at optimum pH of 5. The study on the effect of change in contact time on adsorption of the phenol demonstrated a general increase in the percentage adsorption as shown in Table 4.17 and 4.18. The percentage adsorbent increased from 24.04 % (at 5 minutes) to a maximum of 44.18 % (at 30 minutes).

Table 4.17: Effect of contact time on phenol adsorption (initial concentration (conc) 100 ppm)

Time (min)	Residual conc (ppm)	p-value	%Adsorption	p-value.1
5	75.93±2.66	<0.001	24.07±0.84	<0.001
10	71.09±2.49	<0.001	28.91±1.01	<0.001
15	64.74±2.27	<0.001	35.26±1.23	<0.001
20	58.85±2.06	<0.001	41.15±1.44	<0.001
25	57.91±2.03	<0.001	42.09±1.47	<0.001
30	55.72±1.95	<0.001	44.28±1.55	<0.001
p-value (ANOVA): <0.001				

On the study adsorption of resorcinol from water, percentage adsorption obtained was relatively higher than adsorption of phenol. At 5th minute, adsorption was 35.72 % compared with 24.07 % in case of phenol. As the contact time increased from 25th to 30th minutes rate of adsorption decreased. In resorcinol, gradual increase in percentage adsorption was also observed as contact time increased. Similar increase in rate of adsorption was observed for both phenolic compounds.

Table 4.18: Effect of contact time on resorcinol adsorption (Initial Conc: 100 ppm)

Time (min)	Residual conc (ppm)	p-value	%Adsorption	p-value.1
5	64.28±2.25	<0.001	35.72±1.25	<0.001
10	57.85±2.02	<0.001	42.15±1.48	<0.001
15	48.93±1.71	<0.001	51.07±1.79	<0.001
20	43.61±1.53	<0.001	56.39±1.97	<0.001
25	42.34±1.48	<0.001	57.66±2.02	<0.001
30	37.50±1.31	<0.001	62.50±2.19	<0.001
p-value (ANOVA): <0.001				

It was observed that the rate of adsorption increased significantly from 5 minutes to about 20 minutes. This was explained by presence of high number of active adsorption sites on the maghemite. However, as contact time was increased, the rate of adsorption did not increase significantly due to saturation of active sites. Both systems exhibit pseudo-second-order kinetics. Phenol: 24.07±0.84% to 44.28±1.55% (84% improvement). Resorcinol: 35.72±1.25% to 62.5±2.19% (75% improvement). Equilibrium reached at 25-30 minutes. Resorcinol's higher initial adsorption confirms superior affinity ($p < 0.001$). These results are illustrated further by graph of change in percentage adsorption against contact time in Figure 4.20.

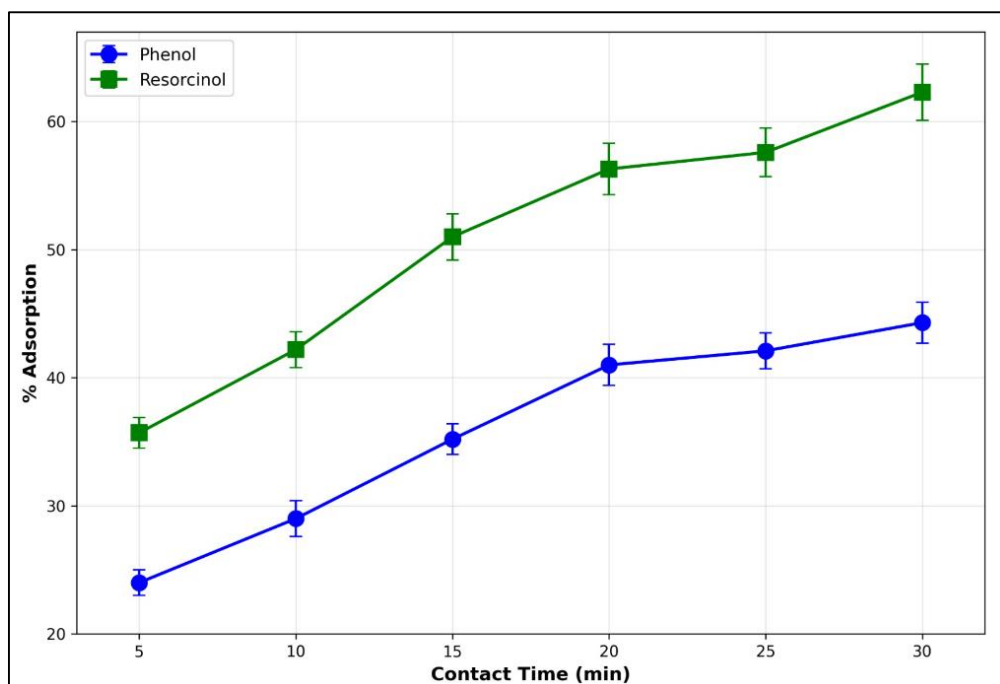


Figure 4.20: Change in percentage adsorption against contact time.

Similar observations were made on study on adsorption of phenol using hematite ($\alpha\text{Fe}_2\text{O}_3$) nanoparticles. The study reported that decrease in rate of adsorption with increase in contact time was attributed to saturation of adsorption active sites of the adsorbent (Dehmani et al., 2020). In another study on removal of methyl blue and methyl green dyes from water using functionalized multiwall carbon nanotubes powered by iron oxide nanoparticles, change in contact time was found to play a major role. When batch adsorption was carried out at varying contact time ranging from 10 to 60 minutes at constant pH of 5.5. It was reported that the rate of adsorption was high for the first 10 minutes. However, the percentage adsorption remains constant there after due to saturation of adsorption sites of the adsorbent (Yentekakis *et al.*, 2025).

In a study of adsorption of Pb^{+2} from water using maghemite-zeolite nano-composite, contact time was found to play a major role in the adsorption efficiency. It was reported to have a 99% adsorption efficiency. Adsorption equilibrium was achieved after 5th minute. After 30th minutes the residual Pb^{+2} in water after adsorption was found to be below detectable levels when analyzed using ICP-AES. After adsorption, the adsorbent was recovered by magnetic separation since maghemite is paramagnetic (Ramos-Guivar *et al.*, 2020).

In another study carried on adsorption of phenol from pharmaceutical waste water. It was found that silver doped magnetite biochar was an effective adsorbent in water remediation. Adsorption efficiency was greatly affected by factors such as change in contact time. When contact time was varied from 150 to 300 minutes, it was reported that rate of adsorption increased with increase in contact time. The highest increase in adsorption occurred between 150th to 180th minutes, then slowed down up to 300th minute due saturation of adsorption sites. Optimum adsorption of 91% was achieved at 300th minute. This observation was attributed to extended time of interaction between adsorbent and adsorbate (Mustapha *et al.*, 2026).

Similar observations were made by Obanla *et al.*, (2023), in this study on adsorption of phenol from aqueous environment using seed shell biochar adsorbent. The study reported that rate of adsorption decreased with increase in contact time. The rate of adsorption was highest at the beginning and gradually decreased with increase in the contact time. This was explained to be caused by the high number of empty adsorption sites at the beginning. Gradual build-up of the phenol on the available site resulted in decrease in the rate of adsorption after some time (Obanla *et al.*, 2023).

4.5.4 Effect of change in temperature on adsorption of phenol using maghemite

During the study, the temperature was varied gradually from 20 °C to 60 °C during adsorption. On analysis, it was found that there was minimal change in percentage adsorption. However, at 20⁰ C the adsorption was 59.72% and reduced slightly to 56.61% at 60 °C as shown by Table 4.19 and 4.20.

Table 4.19: Effect of Temperature on Phenol Adsorption (Initial Conc: 100 ppm)

Temperature (°C)	Residual conc (ppm)	p-value	%Adsorption	p-value.1
20	40.28±1.21	<0.001	59.72±1.79	<0.001
30	41.99±1.26	<0.001	58.01±1.74	<0.001
40	41.21±1.24	<0.001	58.79±1.76	<0.001
50	43.87±1.32	<0.001	56.13±1.68	<0.001
60	43.39±1.30	<0.001	56.61±1.70	<0.001
p-value (ANOVA): <0.001				

Table 4.20: Effect of Temperature on Resorcinol Adsorption (Initial Conc: 100 ppm)

Temperature (°C)	Residual conc (ppm)	p-value	%Adsorption	p-value.1
20	29.63±0.89	<0.001	70.37±2.11	<0.001
30	30.91±0.93	<0.001	69.09±2.07	<0.001
40	39.38±1.18	<0.001	60.62±1.82	<0.001
50	40.06±1.20	<0.001	59.94±1.80	<0.001
60	44.83±1.34	<0.001	55.17±1.66	<0.001

In adsorption of phenol, it was observed a stable adsorption (56-60%) across 20-60 °C with maximum at 20 °C (59.72±1.79%), 3.6% decrease indicates weakly exothermic physical adsorption ($p < 0.001$). in adsorption of resorcinol, it was found to be more temperature-dependent. That is, 70.37±2.11% at 20 °C to 55.17±1.66% at 60 °C (15.2% decrease). Stronger exothermic character indicates more stable surface

complexes ($p < 0.001$). The data on effect of temperature change on the rate of adsorption can be illustrated further by the graph of change in temperature against percentage adsorption in Figure 4.21.

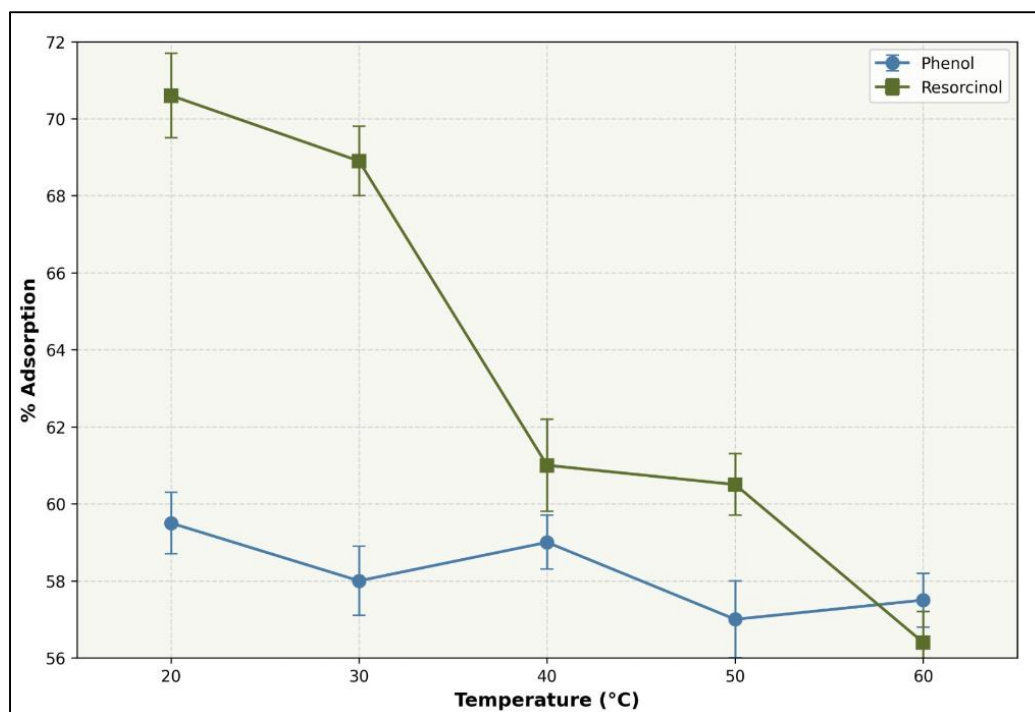


Figure 4.21: Change in percentage adsorption against temperature.

A study on adsorption of methyl blue and methyl green dyes using functionalized multiwall carbon nanotubes powered with iron oxide nanoparticles observed that change in temperature resulted in insignificant effect on the rate of adsorption (Yentekakis *et al.*, 2025). In another study on remediation of pharmaceutical waste waters by removal of phenol using silver doped magnetite biochar, it was found temperature affected rate of adsorption. During batch adsorption, temperature was varied from 30 to 50 °C at a fixed contact time of 300 minutes. The study reported that the highest percentage of 92.9 % was achieved at 30 °C. The rate of adsorption decreased gradually thereafter with increase in temperature. These observations were explained to be caused weakening of the adsorbent-adsorbate bonds with increase in temperature. Therefore, lower temperatures favored adsorption of phenol (Mustapha *et al.*, 2026).

Obanla *et al* (2023) in their study on removal of phenol from aqueous environment using acid-base modified seed shell biochar found that temperature affected the rate

of adsorption. The percentage adsorption decreased with increase in temperature. This was attributed to the exothermic nature of the adsorption process. From calculations it was found that the Gibbs free energy (G^0) decreased as the solution temperature increased denoting that adsorption of phenol becomes less favorable at high temperatures (Obanla *et al.*, 2023).

4.5.5 Adsorption isotherms models for phenolic compounds

Adsorption isotherms are the Mathematical expressions that describe the relation between the quantity of adsorbate on the adsorbent surface (q_e) and the equilibrium concentration in solution (C_e) at a constant temperature are called adsorption isotherm models. These models are essential tools for studying adsorption, as they provide insight into surface characteristics, adsorption capacity, and the adsorbent mechanism. Two common isotherm models have been used in this study to analyze the adsorption of phenol and resorcinol on maghemite: the Langmuir and the Freundlich isotherms.

Both models were applied in their linearized forms, enabling graphical determination of parameters through linear regression. In Langmuir model, a graph of C_e/q_e (y-axis) vs C_e (x-axis) was plotted. The slope gives $1/q_{\max}$ and the y-intercept gives $1/(q_{\max} \cdot KL)$. In Freundlich model a graph of $\log(q_e)$ (y-axis) vs $\log(C_e)$ (x-axis) was plotted. The slope gives $1/n$ and the y-intercept gives $\log(KF)$. The coefficient of determination (R^2) was used to assess the goodness of fit for each model. The model with the higher R^2 value was taken as the best-fitting isotherm for each adsorbate. The equilibrium adsorption capacity (q_e) was calculated using the mass balance Equation 4.9 which gives equilibrium adsorption capacity (Ramos-Guivar *et al.*, 2020).

$$q_e = (C_0 - C_e) \cdot V / m \quad (\text{Equation 4.9})$$

Where C_0 is the initial concentration (mg/L), C_e is the equilibrium concentration (mg/L), V is the volume of solution (L), and m is the mass of adsorbent (g) as illustrated by Table 4.21.

Table 4.21: Comparative summary of adsorption isotherm models

Parameter	Langmuir	Freundlich
Year introduced	1918	1906
Nature	Theoretical	Empirical
Surface type	Homogeneous	Heterogeneous
Adsorption layers	Monolayer only	Multilayer allowed
Maximum capacity	Defined (q _{max})	Not defined
Linear plot	Ce/qe vs Ce	log qe vs log Ce
Key parameters	q _{max} , K _L	K _F , 1/n
Best fit (this study)	Resorcinol (R ² = 0.9967)	Phenol (R ² = 0.9816)
Adsorption mechanism	Monolayer, uniform sites	Multilayer, mixed energies

The Freundlich model fitted phenol adsorption on maghemite best, with $R^2 = 0.9816$, compared to the Langmuir model, with $R^2 = 0.9757$. This suggests that phenol adsorption on a heterogeneous surface occurs via multilayer adsorption. The Langmuir fit of phenol showed a maximum adsorption capacity of 17.38mg/g. The adsorption isotherm summary was given by Table 4.22

Table 4.22: Adsorption isotherm summary

Adsorbate	Best-Fit Model	R ²	q _{max} (mg/g)	Adsorption Type
Phenol	Freundlich	0.9816	17.38	Heterogeneous, multilayer
Resorcinol	Langmuir	0.9967	37.09	Homogeneous, monolayer

In a related study, adsorption of phenol from water using hematite ($\alpha\text{Fe}_2\text{O}_3$) nanoparticles, it was reported that adsorption followed pseudo- second order kinetics. The adsorption fitted Langmuir model. However, in this study adsorption fitted Freundlich model since maghemite was used as adsorbent (da Silva Aires *et al.*, 2024). In adsorption of phenolic compounds from olive oil mill waste waters, it was reported that adsorption followed Freundlich adsorption isotherm where heterogenous layer adsorption occurred (Shmeis *et al.*, 2021).

4.6 Adsorption at optimum conditions

In this section, adsorption had been repeated at optimum conditions. This was done to determine the best percentage adsorption of aluminium ions and phenolic compounds from water using maghemite nanoparticles at optimum conditions of pH, temperature and contact time.

4.6.1 Adsorption of residual aluminium ions using maghemite ($\gamma\text{Fe}_2\text{O}_3$) at optimum conditions

In this study, the test was done five times. Adsorption was done using 0.5 g maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles. Treated de-ionized water samples had initial concentration of 13.948 ppm before adsorption. Adsorption was carried out at optimal conditions reported in chapter 4.4. That is, test was done at pH of 4, temperature was maintained at 65 °C, while contact time was at 75 minutes. After adsorption was done concentration of residual aluminium ions was determined using AAS and the results obtained are as presented by the Table 4.23.

Table 4.23: Percentage adsorption of aluminium ions at optimum conditions

Test	Initial Conc (ppm)	Residual Conc (ppm)	% Adsorption
1	13.948	3.721	73.32%
2	13.948	3.594	74.22%
3	13.948	3.094	77.81%
4	13.948	3.603	74.16%
5	13.948	2.978	78.65%
Mean		3.398	75.63%
SD		±0.337	±2.42%

The results reported the best percentage adsorption of residual aluminium ions at optimum conditions was at 5th test which gave 78.65%. After the 5 repeat tests, an average of 75.63 %. This higher than previous adsorption rates obtain by varying only one of the parameters (pH, temperature and contact time). These results demonstrated effectiveness of maghemite nanoparticle in water remediation by removing residual aluminium ions from water after treatment with Rock Alum coagulants. This concludes that rate of adsorption decreased with increase in pH. The average Al³⁺ Adsorption removal was 75.63% with a low SD of ±2.42%. This demonstrated consistent behavior of the nanoparticles under the optimum conditions.

The results obtained in this study were similar to observations made by Ahmadi *et al* (2017) in study on adsorption of heavy metals using maghemite ($\gamma\text{Fe}_2\text{O}_3$) modified by cross-linked chitosan. Chitosan was modified by linking it with the magnetic iron oxide ($\gamma\text{Fe}_2\text{O}_3$) to improve on its post adsorption separation which if not effectively done would result in secondary pollution. Batch adsorption was carried out at variable physical conditions such as pH and adsorbent dosage in adsorption of Cd⁺² ions from water. It was reported that the highest adsorption occurred at pH 5. It was explained that adsorption decreased with increase in pH due to formation of insoluble metal hydroxides. It was reported the optimum percentage adsorption to be 91% at a contact time of 60 minutes (Ahmadi *et al.*, 2017).

Similar results were reported by Predescu and Nicolae (2012), who found that maghemite nanoparticles were effective adsorbent of Zn, Cu and Cr. The highest

percentage adsorption was obtained at pH 2.5 and contact time of 20 minutes. It was observed that at adsorption of metal ions decreased with increase in pH. This was attributed surface of adsorbent acquiring a positive charge at lower pH, creating high competition for adsorption sites with anions such as hydroxide ions (Predescu & Nicolae, 2012). These results were collaborated by a study in adsorption of copper(ii) ions from water using maghemite. In this study, copper(ii) was adsorbed at constant temperature of 25 °C (Guivar *et al.*, 2017).

In a related study, use of maghemite coated with *Bacillus subtilis* in adsorption of Cd^{+2} from water reported adsorption of 84%. This improved adsorption was achieved at optimum conditions of pH 4, temperature of 30⁰ C and contact time of 1 hour. It was found that metal ions adsorption was improved greatly when in contact with bacterial cell walls coupled with increased surface area of the nanoparticles (Devatha & Shivani, 2020).

4.6.2 Adsorption of phenolic compounds (phenol and resorcinol) using maghemite ($\gamma\text{Fe}_2\text{O}_3$) at optimum conditions

According to the study as per procedure in chapter 3.6, results obtained indicated that on average the optimal adsorption occurred at pH 5, temperature of 20 °C and contact time of 30 minutes using maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles. Both phenol and resorcinol exhibited very close optimal conditions though resorcinol required slightly higher pH. This study had been done by carrying out five tests under similar conditions of pH, temperature and contact time. The concentration of spiked solution prior batch adsorption was 100 ppm. The residual concentration of phenolic compounds after adsorption was determined using UV-Vis in order to calculate the percentage adsorption.

Adsorption of phenol under optimum conditions gave an average of 72.61%, while resorcinol gave slightly higher percentage adsorption of 80.09% as presented by Tables 4.24 and 4.25.

Table 4.24: Percentage adsorption of phenol at optimum conditions

Test	Residual Conc (ppm)	% Adsorption
1	28.24	71.76%
2	25.39	74.61%
3	26.75	73.25%
4	28.91	71.09%
5	27.68	72.32%
Mean	27.39	72.61%
SD	±1.37	±1.37%

Table 4.25: Percentage adsorption of resorcinol at optimum conditions

Test	Residual Conc (ppm)	% Adsorption
1	23.79	76.21%
2	20.24	79.76%
3	17.83	82.17%
4	22.00	78.00%
5	15.68	84.32%
Mean	19.91	80.09%
SD	±3.23	±3.23%

The results in the Tables 4.24 and 4.25 demonstrated that adsorption of phenol was the most consistent with the lowest SD of $\pm 1.37\%$. The mean adsorption of 72.61% indicates moderate to good adsorption. Resorcinol adsorptions had the highest average removal value of 80.09%, which suggests a higher selectivity of the maghemite nanoparticles for resorcinol than for phenol and Al^{3+} . But the highest SD $\pm 3.23\%$, envisaging a slightly higher variability (perhaps due to the experiment). There is a high level of adsorption ($>70\%$) for all the compounds, which suggests that the synthesized maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles are applicable for water treatment.

In a study of adsorption of phenolic compounds using neat clay, it was reported that percentage adsorption of 85% was achieved when the clay was calcinated at 500°C . similar to the results of this study, adsorption of phenolic compounds decreased with

increase in pH. This was explained to be caused by increase in OH^{-1} ions which reduced the interaction of phenolic groups with the adsorbent surface due to competition for active sites. In adsorption of phenol using coconut-based activated carbon, adsorption of over 90% was achieved at pH 7. This is because phenol is a weak acid and anionic form is observed at high pH, while molecular is observed at low pH reducing percentage adsorption at extreme pH environments (Ho, 2022).

Batch adsorption of phenol and 2,4-dinitrophenol using activated carbon from *Tithonia diversifolia* reported efficient adsorption of 99 and 97% respectively. The activated carbon had been activated using KOH and calcinated at 700 °C. Monolayer adsorption (Langmuir adsorption model) was reported. These results correlated with study on adsorption of phenol and resorcinol (Supong *et al.*, 2020). In a related study on removal of phenolic compounds from waste waters using Fe-nano zeolite, similar observations were reported. It was found that the adsorbent gave adsorption efficiency of 63, 70 and 83 % for phenol, 2-chlorophenol and 2-nitrophenol respectively. In addition, the cost of removal of the three phenolic compounds was lower than when activated carbon was used under identical conditions (Tri *et al.*, 2020).

In remediation of water by removal of phenol using silica coated magnetite ($\text{SiO}_2\text{-Fe}_3\text{O}_4$), the nanoparticles were prepared from rice husks which gave silica content of about 18-22%. The magnetite had been prepared by co-precipitation. Adsorption mechanism of phenol involved both cationic and anionic electrostatic attraction with silica coated magnetite. The optimum conditions of adsorption were found to be contact time of 5 minutes at pH of 5 where percentage adsorption ranged from 85 to 92% was achieved. Therefore, magnetic iron oxides such as magnetite and maghemite nanoparticles were found to be efficient and convenient method in water remediation by adsorption of phenolic compounds from water (Wee *et al.*, 2023).

4.7 Chapter summary

This chapter evaluates the effect of use of aluminium coagulant in water treatment and concentration of residual aluminium in treated de-ionized and natural water (river and borehole water samples). treated water was found to have residual aluminium ion (1.9 ppm) higher than recommended level of 0.1 ppm by WHO. Maghemite nanoparticles were successfully synthesized and characterized using XRD and FTIR. The chapter presented and discussed application of maghemite nanoparticles in remediation of

treated water by removal of residual aluminium and phenolic compounds from spiked water samples.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study aimed to remove residual aluminium ions and phenolic compounds from treated water using aluminium based Coagulants using maghemite ($\gamma\text{Fe}_2\text{O}_3$) prepared by green synthesis. From this study, following conclusions were made.

Treatment of water using aluminium based coagulant (Rock Alum) results in elevated levels of residual aluminium ions in treated water. Treated de-ionized water samples gave higher residual aluminium than treated natural water (borehole and river water samples). However, in all treated water samples the levels of residual aluminium ions were higher than recommended levels of 0.1 ppm by world health organization (WHO)

Maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles can be prepared efficiently by green synthesis where iron(iii) salts were oxidized using pineapple (*Ananas comosus*) peels extracts. The maghemite nanoparticles exhibited parameters corresponding to the nanoparticles. Calcination temperature affected the physical properties of the nanoparticles. Moderate calcination temperatures promoted atomic mobility and better structural alignment, resulting in the development of smaller and more homogeneous crystallites.

Maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles are efficient adsorbents of residual aluminium ions from treated water. In adsorption mechanism the Freundlich isotherm provided the best description of aluminum ions adsorption by maghemite nanoparticles, indicating a heterogeneous adsorption process involving multiple binding sites with different affinities. Efficient removal of residual aluminium ions in treated water by adsorption with maghemite ($\gamma\text{Fe}_2\text{O}_3$) nanoparticles is improved by use of low pH for maximum adsorption, increasing adsorbent analyte contact time. However, there was weak correlation reported between adsorption temperature and percentage adsorption.

Maghemite nanoparticles were effectively adsorbs both phenol and resorcinol from water. Other key findings in adsorption of phenolic compounds include; Resorcinol gave superior percentage adsorption of 10-20% higher adsorption than phenol under similar conditions. Optimal adsorption pH for phenol was 5 and 7 for resorcinol. Rate of adsorption of phenol and also resorcinol increased with increase in contact time.

The study found that adsorption of resorcinol was found to be more temperature-sensitive when compared with phenol. Also, the study found that adsorption of phenolic compounds was found to be exothermic, therefore low temperatures favored high percentage adsorption.

Adsorption of aluminium ions at optimum conditions of pH 4 and temperature of 65 °C gave an average of 75.63%. At optimum conditions of pH 5 and temperature of 20 °C, adsorption of phenolic compounds gave 72.61 and 80.09% for phenol and resorcinol respectively.

5.2 Recommendations

From various observations made in the course of this study and also from the results obtained in this study made the following recommendations;

5.2.1 Recommendations for Policy

The study recommends implementation of improvement in procedure that can reduce the levels of residual aluminium ions in water during treatment using aluminium based coagulant can be reduced by: Optimizing coagulation temperature, avoiding use of excessive aluminium-based Coagulants, ensuring effective mixing by agitation at dosing stage, allowing sufficient settlement time for maximum flocculation and efficient flocs filtration.

The study recommends chemical engineers to design an effective water treatment column packed with maghemite nanoparticles for largescale installation in water treatment systems to remediate treated water by removal of residual aluminium ions and phenolic compounds from water meant for domestic use.

5.2.2 Recommendations for Future Work

This study recommends a comprehensive study on development of water treatment coagulants that are not aluminium based. This will ensure water will be clean and safe for human consumption without formation of residual aluminium in the treated water which is harmful to human health.

In this study pineapple peels (*Ananas comosus*) was used as reducing/capping agent. This study recommends future research on use of other plant materials which can be

used as capping agents in green synthesis of nano particles. In addition, the study recommends study on use of other precipitating agent other than sodium hydroxide in synthesis of maghemite ($\gamma\text{Fe}_2\text{O}_3$).

In this research adsorption of residual aluminium was done at different pH, contact time and temperature. The study recommends further investigation on effect of other varying parameters such as change in adsorbent dosage and initial concentration when maghemite($\gamma\text{Fe}_2\text{O}_3$) nanoparticles are used in adsorption of aluminium ions from water. A study on effect of competing ions which could be present, in adsorption of aluminium ions from water was also recommended. This is more pronounced when samples were collected from natural water. The study on adsorption at variable temperature exhibited weak negative correlation. Therefore, there is need to investigate further the dynamics involved

This study recommends future investigation on other factors other than pH, temperature and contact time that affects the adsorption of phenol and resorcinol from water using maghemite ($\gamma\text{Fe}_2\text{O}_3$). In addition, I recommend further investigation on use of maghemite in adsorption of other phenolic compounds such as catechol and halo-benzene in remediation of water.

Using optimum adsorption conditions reported in this study, therefore, recommends investigation of use of maghemite in adsorption of other metallic ions from water. Also, effect of surface modification of maghemite nanoparticles by attachment of functional groups such as carbonyl, nitro, hydroxyl, amino among other should be investigated in future. This should be applied in improving remediation of water by removal of aluminium ions and phenolic compounds.

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APPENDICES

Appendix 1: Research authorization



MURANG'A UNIVERSITY OF TECHNOLOGY

Office of the Director, Postgraduate Studies

E-mail: bps@mut.ac.ke

Website: www.mut.ac.ke

P.O. Box 75-10200

Murang'a, Kenya

Ref: MUT/ARP/PGS/19/2020 /VOL.1

Date: 12th February, 2025

Mr. Daniel Murage – AS501/5001/2023

School of Pure, Applied and Health Sciences,
Murang'a University of Technology,
P.O. Box 75-10200
Murang'a.

Dear Mr. Murage,

RE: APPROVAL OF RESEARCH PROPOSAL AND SUPERVISORS.

I am pleased to inform you that the Directorate of Postgraduate Studies on 10th February 2025 considered and approved your Doctoral research proposal entitled ***"Remediation of Home-Treated water by removal of residual Aluminium and Phenolic Compounds using Hematite $\alpha\text{Fe}_2\text{O}_3$ Nano particles prepared."*** and appointed the following as supervisors:- Murang'a University of Technology

1. Prof. Sylvia Opiyo - Murang'a University of Technology
2. Dr. Peter Walthaka - Murang'a University of Technology
3. Dr. Sharon Kiprotich - Murang'a University of Technology

You may now proceed with your data collection subject to obtaining research permit from NACOSTI, if required. You should also begin consulting your supervisors and submit through them quarterly progress reports to the Director Postgraduate Studies through your CoD and School Dean. Progress Reports forms can be accessed in the Student Portal.

It is the policy and regulations of the University that you observe deadlines. The guidelines on Postgraduate supervision can be accessed in the post graduate Handbook.

Your responsibilities as a student will include, among others;

- i. Maintain regular consultation with your supervisor(s), at least once a month

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- ii. Submit quarterly reports on time, through your supervisors, CoD, Dean and to the Director of Postgraduate Studies;
- iii. Ensure quality work all through;
- iv. Present your research findings at 2-3 seminars/conferences prior to thesis examination.
- v. Publish two articles from your research findings in a refereed journal prior to thesis examination.

For any further clarification please contact the undersigned.

Yours Sincerely,



Prof. Geoffrey Muchiri, PhD
Director, Postgraduate Studies

G.M./MMK



Copy to:

1. Registrar – Academics and Students Affairs
2. Dean, School of Pure, Applied and Health Sciences

Appendix 2: List of publications

This thesis has produced the following papers

Murage, D., & Waithaka, P. N. (2025). Human health effects associated with aluminium poisoning and remediation. *African Journal of Pure and Applied Sciences*, 6(1), 8–13. <https://doi.org/10.33886/ajpas.v6i1.642>

Murage, D. (2026). residual aluminium ions in water treated with “rock alum” coagulants. international journal of research and analytical review. https://www.academia.edu/169547408/residual_aluminium_ions_in_water_treated_with_rock_alum_coagulants