

Remediation of Zn^{2+} ion using Fe_2O_3 impregnated metal oxide nano composite : mechanism, influencing parameters and application

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Technology degree in Chemical Engineering

by

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**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

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This is to certify that this Project Report is the bonafide work of **Godwin Gopinathan (39190005)** and **Dhishon Franklin F (39190003)** who have done the Project work as a team who carried out the project entitled "**Remediation of Zn^{2+} ion using Fe_2O_3 impregnated metal oxide nano composite : mechanism, influencing parameters and application**" under our supervision from (07.09.2022 to 04.04.2023).


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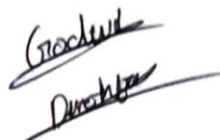

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Declaration

We **Godwin Gopinathan** and **Dhishon Franklin F** hereby declare that the Project Report entitled “ **Remediation of Zn^{2+} ion using Fe_2O_3 impregnated metal oxide nanocomposite: mechanism, influencing parameters and application**” done by me under the guidance of **Dr.D.PRABU.M.S(ByResearch),Ph.D.,** (Internal) and **Dr.K.VIJAI ANAND.M.Sc,Ph.D.,** (External) at Sathyabama Institute Of Science And Technology, Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai, Tamil Nadu is submitted in partial fulfilment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

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ABSTRACT

Water contamination is becoming a major problem for us. It is brought on by a variety of hazardous toxins, including heavy metals, dye, and oil spills. It was determined that treating this was necessary in order to reduce water contamination. The removal of Zn^{2+} using the produced adsorbent is investigated. Zinc nitrate is a very poisonous compound that can lead to cancer of the skin, bladder, and lungs as well as destroy aquatic life. Adsorption is favored over all other removal methods due to its lower chemical consumption and large range of adsorbent options. .Because of its extremely strong magnetism and recyclability, $\text{CaO-Fe}_2\text{O}_3$ has been proven to be useful. The large-scale wastewater treatment facilities, home purifiers, and industrial effluent treatment facilities all employ novel adsorbents with excellent adsorbent capabilities to remove Zn^{2+} . Heavy metals, dyes, and many other contaminants are effectively removed using calcium oxide, which has minimal commercial value. The characteristic of magnetic particles is revealed by a number of characterization techniques, such as SEM with EDX, XRD, FTIR. In the paper below, "Metal oxide helped by activated carbon nanocomposite utilised to eliminate heavy metal ions, an ecological pollutant created by solution," . The metal oxide - AC nanocomposites were distinguished by XRD, SEM-EDX, FTIR. The effectiveness of the nanocomposites as adsorbents to remove heavy metals from natural water sources was investigated. The efficacy of heavy metal purification relies on the pH of the solution, is related to carbon content, the dosage of carbon nanocomposites, and the temperature of the nanocomposites, and decreases with initial concentration of the heavy metals. A cheap, recyclable, scalable, and environmentally acceptable adsorbent particle for extracting heavy metal ions from polluted water was identified as the Metal Oxide-AC. For the removal of heavy metals from wastewater, adsorption methods are widely utilised. Adsorption is a cost-effective method that has gained popularity for its little waste disposal benefit. Before to choosing an adsorbent, consider its affordability, technical suitability, and ease of access to raw materials with minimal detrimental effects on the system. The chapter's originality comes from its coverage of a broad range of adsorbents and how well they remove wastewater that contains heavy metals.

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LIST OF ABBREVIATIONS

ABBREVIATIONS

ACRONYMS

SEM

Scanning Electron Microscopy

FTIR

Fourier Transform Infrared Spectroscopy

EDX

Energy Dispersive X-RAY

XRD

X-RAY Diffraction

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CHAPTER-1 INTRODUCTION

The recent enormous gains in industrialization and urbanization have brought to the forefront, on a global scale, the issue of water pollution as a result of these trends. Significant industrial pollutants consist of dyes and hazardous metals, which are utilised in the production of a wide range of goods, such as textiles, batteries, leather, fertilizer, chemicals, mining, metallurgy, and refining. Other products that contain dyes include leather and textiles. These businesses dump their waste into an environment that already has high concentrations of heavy metal ions and dye, both of which are known to be hazardous to human health. Some of the ailments it can cause include skin irritations and allergies, tumors, headaches, and respiratory problems. It can also cause allergies. Additionally, it is detrimental to aquatic habitats because it reduces the amount of light that can pass through the water. Heavy metals that cannot be broken down by biological processes, such as zinc, cadmium, and copper, nevertheless provide a significant risk to the environment. People should not drink the water since it contains low amounts of heavy metals and other contaminants.[2] They rapidly accumulate inside living organisms, which can lead to infections and problems with metabolism. In addition, the presence of heavy metals poses a significant threat to marine life. Both heavy metals and dye are known to cause substantial damage to ecosystems; hence, it is essential to remove these toxins from sewage in order to safeguard human health as well as the health of the environment.[19] For the purpose of removing dangerous pollutants and heavy metals, numerous treatment processes, such as adsorption, precipitation, membrane filtration, chemical oxidation, ion exchange and electrolysis, are utilised. Methods that fall within this category include those that are physical, chemical, and biological. Chemical modification of AC, which includes impregnation, base, and acid, has demonstrated to be more effective in eliminating heavy metals and dyes from aqueous solutions as compared to earlier treatment procedures. Processing with acid or alkali, as well as the addition of activated carbon with various metal oxides, led to an increase in the adsorption capacity of the material for dye or metal ions.[20] The alteration of surface functional groups was also necessary for the adsorption of certain metal ions. Extensive research has been conducted on the subject of the deposition of heavy metals and colours onto chemically modified

bio-char. It has not been determined whether chemically modified activated carbons are successful at removing heavy metals and effluent dyes by the use of any review studies that have been conducted. This study focuses on determining the most effective treatment methods for increasing adsorption. We also address the amount of dye and heavy metal that is absorbed by activated carbon as a result of the chemical alterations that are made to it. Zinc, cadmium, chromium, and copper are just a few examples of the heavy metals that can be found in waste products from certain industries. When ingested in large quantities, it can cause headaches, nausea, vomiting, liver cirrhosis, renal troubles, difficulty breathing, and stomach pain. It is also a carcinogen. Before being released back into the environment, sewage that contains heavy metals that are toxic to living things must first be treated and purified.[16]. In this setting, the study of nanotechnology has been going on for quite some time. Researchers have also begun using metal oxide nanoparticles as a new generation of adsorbent for the purpose of treating water in order to combat the declining quality of the water supply.[18] Metal oxide possesses a number of useful qualities, including a high surface area to volume ratio, an adjustable surface area, outstanding magnetic properties, bio-compatibility, the ability to be easily separated by an external magnetic field, re-usability, and a relatively affordable price point[21]. As a result, it makes a fantastic adsorbent. In addition, it has been demonstrated that combinations of activated carbon and iron oxide are effective at eliminating a variety of contaminants from aqueous media. According to the research that has been done, the importance of agricultural wastes extends to the fields of science, society, and the economy. This is due to the fact that agricultural wastes are readily available, do not require regeneration, and only require a straightforward treatment before being thrown away. [26]

1.1 HEAVY METALS AND ITS TYPES

1.1.1 ARSENIC (As)

The presence of arsenic can be found in around 95% of all detergents, dyes, paint, metals, semi-conductors, colors, and pharmaceuticals. In addition, the release of increased quantities of arsenic into the environment may be caused by the use of specific herbicides, fertilizers, and methods for feeding animals. Arsenic (As) is a naturally occurring metalloid that is a component of much more than 245 different minerals. It is the 12th most prevalent component in the human body, the 20th most common component in the crust of the Earth, and the 14th most prevalent component in saltwater. Low-level arsenic exposure can cause damage to blood vessels, which can lead to symptoms such as nausea, vomiting, a prickling sensation in the hands and legs, a reduction in the production of erythrocytes and leukocytes, and irregular heartbeats. People can be exposed to arsenic on a chronic basis through a variety of different pathways, including accidental sources, industrial sources, and naturally occurring resources. Because of arsenic pollution, people in every region are now worried about the state of the environment. The health of millions of people is put in jeopardy because of the high amounts of arsenic found in river basins, a topic that has received a lot of attention recently. Arsenic is a semi-metal. In its metallic form, it is brittle, bright, and has a tint that is between silver and grey.[24] Arsenic is a notoriously lethal toxin. Arsenic compounds are frequently employed as rat poisons and insecticides, despite the fact that their use is strictly regulated. When exposed to damp air, the fundamental arsenic will tarnish on its surface. When it is burned in the presence of oxygen, it produces an aroma similar to that of arsenic trioxide and burns with a flame that is bluish-white and very bright. Cancer of the skin is one of the side effects of exposure to heavy metals containing arsenic. Lung and bladder cancer are also possible outcomes of prolonged exposure. The International Agency for Research on Cancer (IARC) has identified arsenic and its compounds as substances that might cause tumors, in addition to advising that people should avoid drinking water that contains arsenic since it is harmful to their health. It is strongly recommended that arsenic concentrations in drinking water not exceed 0.05 mg/L at any point.

1.1.2 LEAD (Pb)

lead is a toxic environmental contaminant. It causes severe damage to a variety of organs in the human body. Lead can sometimes be absorbed through the skin, but the digestive and circulatory systems are often the primary pathways through which it is taken in. Exposure to lead can result in major difficulties with the bronchial system, the genital tract, and the heart due to the metal's high resistivity, per-oxidation, and systemic inflammation. The legal limitations for the quality of water cannot be exceeded when lead ions are present in wastewaters from industrial processes at quantities ranging from 200 to 500 mg/L. Low concentration lead treatments in plants induce severe physiological processes in their capacity for photosynthetic energy, which in turn leads in strong inhibition of plant development. This is because of the tremendous instability in ion absorption that low concentration lead treatments cause. The capacity of lead metal ions to substitute other bivalent (Ca^{2+} , Mg^{2+} , and Fe^{2+}) and mono-valent (Na^{+}) cations is the primary component of the ionic mechanism of lead poisoning, which ultimately has an effect on the cell metabolism of living cells. This effect is ultimately what causes lead poisoning. These processes, along with cell adhesion, inter-cellular and inter-cellular communication, protein folding, maturation, apoptosis, ionic transport, enzyme regulation, and neurotransmitter release, are all influenced by ionic lead poisoning. These are the qualities of their physical appearance. A few of its properties included a low melting point, low strength, an easy manufacturing process, chemical resistance, electrical as well as chemical reactivity with sulfuric acid, chemical resistance in air, water, and soils, and the ability to absorb sound waves, nuclear radiation, and shock waves. The metal lead has a brilliant appearance and a bluish-white tint. It is quite pliable and elastic to the touch. The amounts of lead that are typically found in drinking water are 0.01 mg/L. [22]

1.1.3 MERCURY (Hg)

Mercury on Earth has deteriorated due to the increased sensitivity, quality, and diversity of acute heavy metal poisoning. This change has been for the worse. Mercury, which is a silvery-white metal, will ultimately degrade and solidify into a pliable lump if it is exposed to humid air. It is a neurotoxic molecule that causes damage to micro-tubules and mitochondria, as well as lipid per oxidation and the accumulation of other neurotoxic chemicals. Mercury is released into the environment as a byproduct of activities in a variety of industries, including the pharmaceutical business, the paper and pulp preservatives sector, the agriculture industry, and the industry that produces chlorine and caustic soda. It is recognized as one of the heavy metals that contribute to the degradation of the natural environment. If the kidneys, brain, or developing foetus are exposed to metallic, organic, or inorganic mercury, all three types of mercury can cause damage to those organs. Mercury and other elements, when combined, can combine to generate both organic and inorganic forms of the element mercury. Agriculture, municipal wastewater discharges, industrial sewage discharges, and incineration are the primary human activities that contribute to mercury contamination. Other human activities that contribute to mercury contamination include mining. It is a naturally occurring metal that is odorless, brilliantly white in color, and has no metallic lustre. When heated, it will transform into a gas that has neither a smell nor a colour. Calculations indicate that a total of no more than 2,200 metric tonnes of mercury might be released into the environment every single year. Organic mercury poisoning can be difficult to detect since its symptoms, such as stress, headaches, tremors, weariness, hair loss, and memory issues, are similar to those of other illnesses. These symptoms include stress, headaches, tremors, exhaustion, and hair loss. It has a significant potential for bioaccumulation and is hazardous in its natural state. There can be no more than 0.001mg per litre (mg/L) of mercury in water that is suitable for human consumption. [10]

1.1.4 CADMIUM (Cd)

Cadmium has emerged as a key metal that is employed in the production of nickel-cadmium (Ni-Cd) rechargeable batteries as well as an anti corrosion coating for steel and iron. Cadmium has also found use in the medical field.[25] During World War I, cadmium was utilised for the very first time in the place of tin in the manufacturing of paint as well as in the capacity of a tint. In modern times, it can be discovered in the production of several goods, including tobacco smoke, rechargeable batteries, some types of alloys, and more. Cadmium is utilised in numerous products manufactured in the current day, including batteries, coatings, alloys, solar cells, pigments, plastic stabilizers, and coverings (electroplating). It is also utilised in the production of plastic stabiliser. It has been demonstrated that cadmium can be found in seafood, wheat, and rice. Cadmium can also be found in some drinking water. The lungs are cleaned of cadmium by breathing in dust from commercial settings. In industrial settings, being exposed to cadmium, whether for a short period of time or for a longer period of time, can be harmful to the kidneys and induce lung illness. The levels of cadmium in the blood of smokers are approximately twice as high as those of non-smokers. Cadmium levels in drinking water are allowed to reach a maximum of 0.03 mg/L.[14]

1.1.5 CHROMIUM (Cr)

The crust of the earth and the ocean are both sources of chromium (Cr), a naturally occurring heavy metal that is utilised in various manufacturing processes. The most common and stable forms of chromium have been determined to be trivalent and hexavalent, with oxidation states ranging from +2 to +6. The name "chromium," which derives from the Greek word *chromes*, which means "color," alludes to the toxic hues that can appear in a range of intensities. The word "chromium" originates from the Greek word *chromes*. Chromium (Cr) is an example of a complex ion that is part of Group VI of the Periodic Table. It increases an alloy's resistance to corrosion and lengthens its durability. Chromium is a metal that is difficult, brittle, and shiny. It has a tint similar to silver grey and is very easy to polish. Because it produces and develops green chromic oxide when it is heated, it does not oxidize when it is exposed to air. Due to the metal's

sensitivity to oxygen, chromium soon produces a thin oxide coating that acts as a shield and protects it from the element. Heavy metals can accumulate in the body when chromium is present, which can lead to a variety of health problems. Hypothyroidism is a condition that can impact a person's skin, kidneys, neurological system, and digestive system, among other body systems. Other disorders that impact these systems include the development of many cancers in the lungs, throat, stomach, liver, testicles, and spine. Other diseases that affect these systems include diabetes and heart disease. The maximum chromium concentration that can be present in drinking water is 0.1 mg/L.[15]

1.1.6 COPPER (Cu)

Copper (Cu) is an element that occurs naturally and is characterized by a reddish-brown colour and a high degree of malleability in its purest form. The most common applications for copper can be found in industrial machinery, roofing, plumbing, and electrical wiring. The vast majority of these applications require the usage of copper in its purest form. However, in order to attain higher degrees of hardness when it is required to do so, it can be alloyed with a number of other metals. Heavy metals are known to have deleterious effects on a range of human organs, both in the short term and over the long term. The harmful effects of heavy metals can lead to a number of health problems, such as cancer, genetic illnesses, issues with the immune system, digestive and urinary issues, problems with the central nervous system, lesions, ischaemia, and ulcers. Copper has the ability to both speed up and slow down the oxidation process.[4] Copper plays a significant role in the human metabolism because it assists in the normal functioning of a wide variety of fundamental and vital enzymes and contributes to the creation of haemoglobin, myelin, and melanin.[7] Copper also helps regulate blood sugar levels. It is necessary for the bio-molecules structural as well as metabolic co-factors in order for bio-molecules to function properly. As a transition metal that signals both within and outside of living cells, as well as among live cells themselves, copper plays an essential part in the transmission of information both inside and outside of the brain. The recovery of heavy metals in general, and copper in particular, is a topic that is receiving a lot of attention from the general public because of the positive effects that removing pollutants from industrial

effluents may have on the environment and the economy. The maximum allowable amount of copper that can be found in drinking water is now 1.3 mg/L.

1.1.7 NICKEL (Ni)

Nickel, a brittle and silvery-white metal, can solidify into formations resembling cubes. It is also resistant to corrosion and features ductility and malleability in addition to high strength. In point of fact, researchers have discovered that each cigarette contains anywhere from 1.1 to 3.1 g of nickel. It is possible that this metal is present in the smoke in the form of nickel carbonyl, which is exceedingly detrimental to one's health. Nickel can also enter individuals' bodies through the consumption of certain foods. In point of fact, a wide variety of foods, such as cocoa, chocolate, and almonds, as well as spinach, asparagus, carrots, broccoli, and green beans, contain considerable amounts of this potentially harmful element. Nickel is mistakenly consumed when food is cooked on stainless steel appliances or when food comes into contact with the mouth. In addition, nickel can be absorbed via the skin. Nickel and nickel compounds are common examples of hazardous substances that can be found in industry. It is well known that nickel and nickel compounds can have an effect on people even when they are not exposed to them in the workplace. Because it is found in air, food, and water, nickel is accessible to the whole population and can be absorbed by them. Regarding the potential adverse health effects of occupational nickel exposure, inhalation is a significant route that must be considered. Even though the majority of nickel found in the human body comes through food and drink, the inability of the intestine to absorb nickel makes this route far less important. Those who are subjected to nickel may experience a variety of adverse health impacts, including allergies, conditions affecting the kidneys, heart, and lungs, fibrosis, and cancers of the lung and nose. The maximum concentration of nickel that can be found in drinking water is 0.1 mg/L.

1.1.8 ZINC (Zn)

Zinc is a metal that has a dazzling bluish-white appearance. The element can be found in the periodic table's Group II (b) section. When it is at normal temperature, the material is crystalline and brittle, but when it is heated, it changes into a substance that is ductile and malleable. Zinc (Zn), one of the most frequent heavy metals in the Earth's crust, is regarded to be an essential compound metal necessary for the growth of living things. This is due to the fact that zinc is a component of a large number of major proteins and governs the regulation of a number of immune-modulatory processes. Zinc is also one of the most common heavy metals in the Earth's crust. However, its toxicity, which will be maintained by a number of human sources, ongoing exposure to contaminated places, and other natural occurrences, parallels its necessity. The equilibrium between dietary zinc absorption and zinc loss from the body is referred to as zinc's homeostasis, and zinc is an essential heavy metal for human health. Zinc is essential to the functioning of many different biological processes, including those that ensure survival, promote growth, and heal damaged tissue. Due to the high bio availability of zinc found in a variety of fruits, vegetables, and dairy products, these foods are considered to be good sources of zinc for human consumption. The most common applications for zinc metal are in the production of brasses and alloys for die casting, as well as the prevention of corrosion in iron and steel. Zinc is more resistant to common atmospheres than iron is, and as a result, it corrodes at a significantly slower rate. Symptoms of heavy metal poisoning in zinc include nausea, vomiting, and diarrhoea. Iron, on the other hand, is more susceptible to corrosion in typical environments. An overdose of zinc can cause a variety of adverse health effects, including metallic oxide fever, digestive and liver problems, and more. The maximum allowable level of zinc in drinking water is 5.0 mg/L.

1.2 DIFFERENT TYPES OF MAGNETIC NANOPARTICLES.

1.2.1 Carbon-based nanoparticles as their primary component

The ability of various carbonaceous compounds, such as carbon nanotubes, carbon black, graphene oxide, and bio-char, to assimilate various environmental toxins has been the subject of extensive research in recent years. In the treatment of wastewater and water, activated carbon is the type of carbon adsorbent that is most commonly used.[3]. For example, the removal of heavy metals from aqueous solutions is one of the many environmental applications that can benefit from the production of activated carbon in the form of sorbents in a variety of forms. A new innovation in the study of heavy metal adsorption calls for the use of bio-char in place of activated carbon derived from coal. This change was brought about by the significant increase in manufacturing costs that are associated with the traditional approach. Since the development of nanotechnology, a great number of research have been carried out on the potential applications of graphene-based compounds, such as Carbon Nanotubes, reduced Graphene Oxide, and Graphene Oxides, for the removal of hazardous metals.

1.2.2 Nano zero valent ion

Nano scale zero-valent nanoparticles have emerged as an innovative and extremely effective technology for the remediation of heavy metals.[6] This is due to the vast specific surface area that these nanoparticles possess as well as their outstanding capacity for reduction. Because of its great reactivity, extraordinary reactive longevity, and low toxicity, nano scale zero-valent ion has shown exceptional promise for the reduction of heavy metal ions in aqueous solutions. has been utilised successfully to treat a range of metallic ions in aqueous solutions.[8]

1.2.3 Silica based nanoparticles

Nanomaterials have been the subject of investigation and study by scientists throughout the past decade. SiO_2 has long been regarded as the best material for water treatment due to its unique qualities, which also include financial viability, non-toxicity, great acid tolerance, and ease of modification. Several nano structured silica materials have been utilised to remove heavy metals. SiO_2 has long been regarded as the finest material for water treatment.[11]. The improved performance in surface group absorption characteristics of SiO_2 was the focus of the initial investigations, and the effect of group type on the sorption of active SiO_2 has been studied only on occasion. It should be noted that the precise amounts of complex chemicals bound to these, as was stated earlier, modified SiO_2 nanoparticles are unknown. This is because the precise amounts of complex chemicals bound to these nanoparticles have not been studied. As a consequence of this, it may be difficult, if not impossible, to identify with absolute certainty whether or not particular chemical groups have a higher affinity for the target ion in question.

1.2.4 Nanocomposites

removing impurities from water by utilising nano structured materials. They feature a high ratio of surface area to volume, which contributes to their excellent attributes, as well as their compact nature, which gives them a huge surface area. These qualities contribute to an increase in the efficiency with which nano structured materials can be removed. These materials are distinguished from other types of materials because to the extraordinary features they possess, which include a huge specific surface area, a significant level of reactivity, and the capacity to catalyse reactions.[9] Nano-structured particles, because of their high surface area, have a great number of active sites that are capable of reacting with a wide variety of chemical species. There has been a recent uptick in interest in nanocomposite materials, which are produced by combining nano structured materials with the inter-layer space of clay particles. This is because nanocomposite materials possess distinctive physicochemical features and have

the potential to find uses in a wide range of industries.[17] The excellent surface qualities of nano-silica were put to use in the production of two innovative composites via a chemical procedure that was straightforward and safe for the surrounding environment. The nanocomposite was evaluated using a variety of different approaches in order to provide evidence for the shift. The nanocomposite was utilised to either absorb or eliminate metals after first being positioned into a glass column.[12]

1.2.5 Nanoparticles Comprised of Polymeric Substances

Over the course of the last 10 years, metallic nanoparticles adsorption (MNP) has received a great deal of attention as a result of its efficiency as well as the extensive number of uses it might have. Because carrying out the treatment won't require much effort on your part. Surface polymeric MNPs, which includes such MNP polymer composites with core-shell nanoparticles, have recently attracted a great deal of interest. This is due to the fact that the polymer shell prevents the core portion of the Magnetite nanoparticles from collapsing into bulk materials and improves the dispersion stability of the core-shell nanoparticles in a suspension medium. The organic and aqueous phases are both capable of successfully distributing these polymeric nanoparticles. It is possible to speedily synthesize polymeric nanoparticles by employing a straightforward precipitation method and low-cost starting ingredients.[23]

1.2.6 Ceramic Nanoparticles

Ceramic membranes are becoming increasingly used in a wide number of applications due to the large chemical and temperature operating range that they offer. The robustness of the ceramic membrane is an important consideration throughout the application process. It has been exploited in the process of oil extraction from waterways as well as the cleaning of effluent from the dyeing process. Ceramic membranes' remarkable anti-fouling properties and long operational life greatly minimize the high startup design complexity. A new area of research that uses a ceramic barrier for the removal of pollutants has very few published studies. Ceramic water filters are utilised in over 20 countries as an

alternative point-of-use water purification system. Although ceramic membranes have a number of benefits, their usage in large-scale applications for metallic ion-contaminated effluent is limited because of the greater cost of membrane fabrication. Ceramic membranes have been shown to be effective at removing metallic ions from water. Clay and trash are the two materials that are gathered from the surrounding area and used in the manufacturing of ceramic water filters. In comparison to polymeric composite membranes, ceramic composite membranes have superior anti-fouling properties and greater mechanical and chemical durability. It has been demonstrated that CWFs can effectively eradicate viruses larger than 1m in diameter by removing nearly 99% of particles larger than 1m in diameter. Ceramic composite membranes also have superior anti-fouling properties than polymeric composite membranes.

1.2.7 Metal Oxide Nanoparticles

According to research, surface effects have a significant impact on the magnetic properties of iron oxide nanoparticles [1]. Fe_3O_4 nanoparticles (MNPs) are a new class of nanomaterials that have the potential to be used in environmental cleaning. Iron-based magnetic nanoparticles have distinctive characteristics, such as a greater surface-to-volume ratio, a reduced demand for chemicals, and the absence of secondary pollutants. Direct silylation of magnetic particles in order to produce Fe_3O_4 adsorbents is accomplished through the use of cross-linking agents, organic gas precipitation, polymer coating, and surfactant adsorption the vast majority of the time. The scientific community has shown a significant amount of interest in materials that have nanoscale-specific features. Fe_3O_4 nanoparticles are an intriguing new category of nanometer-sized components because they can exhibit a variety of distinct and specified properties.[13] These properties include a large surface area, the possibility for self-assembly, high precision, chemical sensitivity, catalyst potential, and the absence of internal diffusion resistance. These properties hold great promise for the development of new or improved wastewater treatment innovations. Because of their super paramagnetic nature, these particles are drawn to a magnetic field; however, once the field is removed, they lose all of their magnetic qualities. Magnets can be used to remove pollutants,

and the super-para magnetism supplied by Fe_3O_4 nanoparticles is an added benefit.[5]

1.3 HEAVY METAL REMOVAL TECHNOLOGY

The dangers of having heavy metals in the food chain as well as within our bodies have been discussed in greater depth in the portions of the thesis that came before this one. Because of this, it is of the utmost importance to treat wastewater and remove as many heavy metals as is reasonably practicable from it before releasing the wastewater back into the environment.[30] Heavy metals have risen to the top of the list of most concerning environmental pollutants over the course of the last few decades as a result of the development of more stringent rules and regulations governing the treatment and disposal of wastewater. As a result, the technologies that can remove heavy metals from wastewater are of considerable interest and significance.[27]

1.4 THE RELEASE OF CHEMICALS

Chemical precipitation is the removal method that is used most frequently in wastewater treatment plants. This is primarily due to the ease with which it can be carried out. The solubility of the metals involved, the pH of the wastewater, the concentration of the metals in the produced wastewater, and the presence of a variety of other substances can all have an effect on the precipitation factor of the metal. Precipitation can also be affected by the concentration of the metals in the produced wastewater. Depending on the chemicals that were used for the precipitation process, a utilised chemical reacts with the ions of heavy metals that are present in the effluent to generate insoluble precipitates of the heavy metals. These precipitates can take the form of hydroxide, carbonate, sulphide, or phosphate. The generated precipitates are subsequently removed as residue after going through the processes of coagulation, sedimentation, and filtration. Alterations in parameters like pH, temperature, conductivity, and metal concentrations can be utilised to improve the rate at which metal ions are removed from a solution. The substantial quantity of concentrated and poisonous sludge that is created necessitates extra treatment as well as disposal. Despite the fact

that the procedure is very basic, effective, and can be cost-effective when compared to alternative removal technologies, the disposal and treatment of the produced sludge is necessary.

1.5 ION EXCHANGE

The process of eliminating heavy metals through ion exchange is one of the more easy options available. The process, as its name suggests, involves exchanging the metal ions that are present in waste water with the ions that are present in an ion exchanger that is either synthetic or natural. Zeolites are the natural ion exchangers that are used more frequently than any other type. Synthetic ion exchangers are favoured over natural ones due to the former's superior efficiency and the latter's ability to extract the vast majority of the targeted metal ions from the latter's solution. The positively charged, potentially toxic metal cations that are affecting the purity of the water, such as zinc, nickel, and copper ions, are exchanged with the positively charged ions from the resins, such as hydrogen and sodium ions, in equivalent amounts in the solution. The same is true for the negatively charged unwanted anions that are present in the wastewater solution, such as hydroxyl anions. Ion exchangers are insoluble solid substances. The polymers can be categorized as either cation or anion exchangers, and this designation is determined by the charges of the ions that are being traded. The approach is particularly sensitive to a variety of parameters, such as pH and temperature, as well as the concentration and duration of contact with the metal. Ion exchange procedures are only suitable for the treatment of heavy metals at low concentrations. Despite their many benefits, including excellent treatment and removal efficiency as well as cost effectiveness (low cost and operating materials), ion exchange methods cannot treat higher concentrations of heavy metals.[29]

1.6 ADSORPTION

The remediation of wastewater that is contaminated with heavy metals also makes use of a removal process called adsorption. Adsorption is the process that takes place when the solute in the liquid phase of polluted effluent accumulates on solid or liquid absorbents to form a molecular film. This can take place on either solid or liquid absorbents. In order to successfully remove metals from effluent water, numerous absorbents, such as activated carbon, carbon nanotubes, metal oxides, and a wide variety of agricultural and bio absorbents, have been demonstrated to be effective. Activated carbon is still the most common type of absorbent due to the fact that its removal rate of some metal ions is greater than 99.9%. Activated carbon is an effective method for eliminating heavy metals; nevertheless, researchers are constantly working to bring down the cost of the process. Because commercial carbon sources are dwindling, the price of carbon absorbents is climbing every day, and activated carbon materials cannot be recycled, it is essential to find substitutes that do not affect the quality of the final product. Because of its low cost and its ability to treat metal-contaminated wastewater in a natural way, bio adsorption is quickly becoming an increasingly popular treatment method. It has been established that bio adsorbents, which can be either living or dead biomass, can remove heavy metals from wastewater and sludge.[28] These adsorbents can be obtained for a low cost and are easily available. In general, there are three types of sources for biomass: 1) non-living biomass, 2) biomass derived from algae, and 3) biomass derived from microbes. Because of its widespread availability, high treatment efficiency, low cost, and high metal sorption capacity, algal biomass has become the most popular option out of the three, and it is also the one that is now garnering the most attention.

1.7 APPLICATIONS OF MAGNETIC NANOPARTICLES

- ❖ Industrial applications
- ❖ Medical and Biomedical applications
- ❖ Catalysis
- ❖ Environmental usage
- ❖ Molecular Imaging
- ❖ Magnetic Separation

1.8 CHARACTERIZATION OF MAGNETIC NANOPARTICLES

- ❖ Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy (SEM-EDX)
- ❖ Fourier Transform Infrared Spectroscopy (FTIR)
- ❖ X-Ray Diffraction (XRD)
- ❖ Vibrating Sample Magnetometer (VSM)
- ❖ Transmission Electron Spectroscopy (TEM)
- ❖ Braunauer-Emmett-Teller (BET)
- ❖ Atomic Absorption Spectroscopy (AAS)

CHAPTER-2 LITERATURE SURVEY

“A Review on Preparation of Activated Carbon from Coconut Shell”

Prof Rangari P.J, Priyanka Chavan.

Activated carbon is broadly defined to include a wide range of amorphous carbon based materials prepared in such a way that exhibit a high degree of porosity and an extended surface area. For many centuries the activated carbon was used in the form of carbonized wood. The earliest known use of carbon in the form of wood chars by the Egyptians and Sumerians was in 3750 BC for the reduction of ores in the manufacturing of bronze, domestic smokeless fuel and medicinal application. In 1500 BC, Egyptian papyrus was used as adsorbent for odorous vapors from putrefying wounds and from within the abdominal tract. The wrecks of Phoenician trading ships suggest that drinking water was stored in charred wooden barrels. This practice was certainly still in use in the 18th century for extending the use of potable water on long sea voyages. This Paper deals with an extensive literature survey on the present research work. This literature search provides a background and guide for the entire study. This Paper includes the review on historical appraisal of activated carbon, activated carbon preparation methods, application of activated carbon, wastewater treatment methods, classification of textile dyes and effect of process parameters on activated carbon preparation and adsorption studies.

“The Potential of Activated Carbon in the Treatment of Water for Human Consumption, a Study of the State of the Art and Its Techniques Used for Its Development “

Adenes Teixeira Alves^{1*}, Dimas José Lasmar¹, Ires Paula de Andrade Miranda², Jamal da Silva Charr¹, Jardson dos Santos Reis¹.

This review article addresses the use of biological activated carbon in water treatment and its techniques for its development, with the premise of analyzing the potential of Activated Carbon (AC) in the treatment of water for human consumption. For this end, it aims to identify the techniques used for the production of AC, use and its benefits, production from vegetable waste for water treatment. Activated carbon has the ability to selectively collect gases, liquids and

impurities inside its pores, which is the reason it is widely used in filtration systems and for that the treatment of water, the coal fulfills the function of adsorbent, retaining in its pores certain types of impurities: large particles that cause undesirable color, taste or odor in the water. In the treatment of effluents, coal is used for clarification, deodorization and purification of effluents.

“Waste water treatment by removal of heavy metals using EDTA functionalized chitosan-graphene oxide nanocomposites”

Asha Radhakrishnan^{1*}, Arya Sugathan², Deepa Vijayan², B. Beena³

This study focuses on the preparation of Ethylenediaminetetraacetic acid (EDTA)-functionalized magnetic chitosan (CS) graphene oxide (GO) nanocomposites (EDTA-MCS/GO) by biogenic method and its removal efficiency for heavy metals namely, Pb(II) and As(III) from aqueous solutions. The synthesized nanocomposite was characterized by XRD, SEM, and FTIR analysis. The influence of various operating parameters, such as pH, metal ion concentration, and contact time on the removal of the metal ions, was investigated. The equilibrium data was evaluated by Langmuir, Freundlich and sips isotherms, while the heavy metal adsorption reaction kinetics was analyzed by Lagergren pseudo-first-order and pseudo-second-order kinetic models. The adsorption–desorption studies conducted over 6 cycles illustrate the viability and repeated use of the adsorbent for the removal of Pb(II) and As(III) from aqueous solutions.

“Nanomaterials for the Removal of Heavy Metals from Wastewater”

Jinyue Yang, Baohong Hou *, Jingkang Wang, Beiqian Tian, Jingtao Bi , Na Wang, Xin Li and Xin Huang *

Removal of contaminants in wastewater, such as heavy metals, has become a severe problem in the world. Numerous technologies have been developed to deal with this problem. As an emerging technology, nanotechnology has been gaining increasing interest and many nanomaterials have been developed to remove heavy metals from polluted water, due to their excellent features resulting from the nanometre effect. In this work, novel nanomaterials, including carbon-based nanomaterials, zero-valent metal, metal-oxide based nanomaterials, and nanocomposites, and their applications for the removal of heavy metal ions from wastewater were systematically reviewed. Their efficiency, limitations, and

advantages were compared and discussed. Furthermore, the promising perspective of nanomaterials in environmental applications was also discussed and potential directions for future work were suggested.

“The role of some important metal oxide nanoparticles for wastewater and antibacterial applications: A review”

Taiba Naseem, Tayyiba Durrani

Nanotechnology has great potential to improve water purification and decontamination efficiency. Nanomaterials are efficient at removing organic and inorganic pollutants and heavy metals from wastewater and killing microorganisms. The distinctive characteristics of metal oxides make them the most varied class of materials, and they have properties that cover almost all aspects of solid-state physics and material science. Only a few review articles in the literature specifically address metal oxide nanoparticles and their application in the treatment of wastewater. This review article aims to fill this gap and present a thorough review of the various types of metal oxide nanoparticles in contaminating water impurities. Metal oxide nanoparticles have great potential for treating contaminated water due to their unique properties, such as their large surface areas and low concentration. This paper discussed in detail five metal oxide nanoparticles and their applications for antibacterial and wastewater applications.

“Metal Oxide Polymer Nanocomposites in Water Treatments”

Francis Opoku, Ephraim M. Kiarii, Penny P. Govender and Messai Adenew Mamo

Recently, several pollutants such as dyes, pharmaceuticals and phenolic compounds, which can cause toxic effects to human health, have identified in water resources. Water pollution has extensively studied and several conventional techniques, such as chemical treatment, adsorption, biological treatment, and membrane-based separation, have adopted for pollutants removal from wastewater/ water resources. However, these techniques had led to the production of soluble refractory organic compounds and health threatening bacteria that are hard to be removed. Recently, photo-catalysis has considered as one of the most viable technology for water treatment using sunlight to eliminate harmful bacteria and pollutants owing to its cost-effectiveness and high efficiency.

Metal oxide and polymers have become promising materials for water treatment owing to their properties, such as surface mobility, large surface area and superb magnetic and optical properties. This book chapter discusses recent design and synthesis of visible light response polymer/metal oxide nanocomposite through several synthetic strategies for water treatment. The results show that the polymer-metal oxide nanocomposite possesses a superior photo degradation activity toward pollutants under simulated visible light. Major challenges in polymer-metal oxide nanocomposite synthesis and future research perspectives for developing alternate synthesis methodologies are also discussed.

“Metal oxide-cellulose nanocomposites for the removal of toxic metals and dyes from wastewater” Opeyemi A. Oyewo , Elias E. Elemike , Damian C. Onwudiwe , Maurice S. Onyango

Waste water remediation of toxic metals and dyes could be complex to achieve owing to several factors. Various techniques have been developed for the removal of these pollutants. However, the success of these water treatment techniques mainly depends on the choice of appropriate media. Most of the commercial and chemically synthesized materials for water treatment create additional hazards due to the leaching of toxic chemicals into the process water. Consequently, studies on the development of better materials which could achieve high efficiency without posing any health risk led to the exploration of non-toxic composite materials such as those composed of cellulose and metal oxides. The focus is on the choice of materials and the best methods that allows their combination in order to address the pollution caused by heavy metals and dyes. This review critically analyses or assess the use of cellulose incorporated with metal oxides in the removal of toxic metals and dyes from industrial effluent. The nanocomposites have been accepted as a technique that circumvents the problems associated with the use of only metal oxides in water purification. They provide a platform for an improvement in the hydrophobicity of the composite with concomitant efficiency in adsorption and photo-catalysis.

“A review on transition metal oxides based nanocomposites, their synthesis techniques, different morphologies and potential applications” S Yadav , N Rani

In the field of nanotechnology and nano-science, transition metal oxides based nanocomposites (TMONCs) are promising for various application uses such as Supercapacitors, Sensors, Bactericidal properties, Photocatalytic Degradation, Solar Cells etc. Modification of transition metal oxide nanoparticles (TMONPs) to TMONCs by doping/mixing of another transition metal and metal oxide, carbon based nanoparticles, conducting polymers etc. to achieve enhanced surface area, increasing surface activities or number of active surface sites, reducing electron-hole recombination, increasing charge transfer processes etc. have been reported in literature. These improved properties are the possible reason for the enhancement in its practical applications efficiencies. This review summarizes recent development on transition metal oxides based nanocomposites for different potential applications. Also synthesis methods of transition metal oxide based nanocomposites have obtained an increasing attractions to achieve cost effectiveness and environment friendly routes of synthesis with high rate of production, high yield of product and also less toxic waste production. Transition metal oxides nanocomposites have been fabricated by various methods such as Microwave assisted synthesis technique, Sol-Gel method, Biosynthesis method, Co-precipitation process, Simple Chemical method etc. Different morphologies of transition metal oxides based nanocomposites have been summarized in this review article. Herein, this paper discuss about several reported synthesis techniques, various characterization techniques used for structural and surface properties identifications, different morphologies and various potential applications of transition metal oxide based nanocomposites.

“Impact of metallic and metal oxide nanoparticles on wastewater treatment and anaerobic digestion” Yu Yang, Chiqian Zhang and Zhiqiang Hu*

Metallic and metal oxide nanomaterials have been increasingly used in consumer products (e.g. sunscreen, socks), the medical and electronic industries, and environmental remediation. Many of them ultimately enter wastewater treatment plants (WWTPs) or landfills. This review paper discusses the fate and potential effects of four types of nanoparticles, namely, silver nanoparticles (AgNPs), nano ZnO, nano TiO₂, and nano zero valent iron (NZVI), on waste/wastewater treatment and anaerobic digestion. The stability and chemical properties of these nanoparticles (NPs) result in significant differences in antimicrobial activities.

Analysis of published data of metallic and metal oxide NPs suggests that oxygen is often a prerequisite for the generation of reactive oxygen species (ROS) for AgNPs and NZVI, while illumination is necessary for ROS generation for nano TiO₂ and nano ZnO. Furthermore, such nanoparticles are capable of being oxidized or dissolved in water and can release metal ions, leading to metal toxicity. Therefore, AgNPs and nano TiO₂ are chemically stable NPs that have no adverse effects on microbes under anaerobic conditions. Although the toxicity of nanomaterials has been studied intensively under aerobic conditions, more research is needed to address their fate in anaerobic waste/ wastewater treatment systems and their long-term effects on the environment.

Dr.D.Prabhuet al. (2021)has published a scientific article titled ***“Feasibility of Magnetic Nano Adsorbent impregnated with activated carbon from animal bone waste”***.

Chromium is a heavy metal that causes a lot of pain for the environmental elements when it is present in effluents. This MNA(Magnetic Nano Adsorbent) from animal wastes is used to remove heavy metals like chromium. The new MNA Fe₃O₄-BAC was created, examined, and employed in the study as an effective sorbent for the elimination of chromium in liquid solution. The thermally treated BAC is co-precipitated onto the magnetic nano adsorbent under ultrasonic conditions. The Fe₃O₄-BAC's enhanced shape is seen in the SEM imaging with enhanced morphology between 100 and 446 nm, and narrow size distribution nanoparticles. At an acidic pH during a contact period of 60 minutes with an adsorbent concentration of 5g/L, optimal adsorption has been discovered. The optimum adsorption rate of nano-adsorbent is 27.86 mg/g. The application feasibility has been confirmed by thermodynamic and isotherm experiments. Additionally, the kinetic experiments demonstrate the examined Fe₃O₄-BAC's propensity towards pseudo-first order models.As a result, Fe₃O₄-BAC may have use to remove chromium in aqueous systems as an adsorbent.

Fawzia I. El-Dibet al. (2020)has published a journal on the topic of ***“Study the adsorption properties of magnetite nanoparticles in the presence of different synthesized surfactants for heavy metal ions removal”***.

Co-precipitation is used to create nanomaterials (Fe_3O_4), which are then maintained by 2 non-ionic and one cationic surfactant. The chemical composition of the produced surfactants was confirmed by ^1H NMR. Fourier transform infrared spectroscopy (FTIR), size distribution (DLS), and magnetic characteristics are used to analyze the produced magnetite nanoparticles (magnetic susceptibility analysis). The produced magnetite nanoparticles were shown by monoclinic crystal structure according to XRD. The magnetic nanoparticles were equally dispersed, as shown in images captured using a (TEM). Using atomic absorption spectroscopy, the produced nanoparticles' ability to adsorb lead, zinc, and cadmium metal ions was calculated (AAS). The pH of the solutions containing metal ions and adsorbent dosages are just a couple of the variables influencing the uptake improvement studies of the produced nanoparticles.

Christos Liosiset al. (2021) has published an article titled ***“Heavy Metal Adsorption Using Magnetic Nanoparticles for Water Purification: A Critical Review.”***

A framework for assessing the important findings of current studies on metal removal using MNP is presented with an emphasis on water quality and technology applicability. Numerous studies have been conducted with an emphasis on the synthesis, characterization, and ability to absorb bare & modified both magnetic nano-structures and the related desorption process of the approaches. The experimental techniques exhibit strong adsorption capability for contaminants from aquatic solutions, according to the current review study. Additionally, recycling the nanoparticles used up to five times results in an efficiency of up to 90%. Additionally, it should be noted that nanoparticles have occasionally been recycled up to 22 times.

Esmaeel Darezereshkiet al. (2018) has published scientific article titled ***“Influence of heavy metals on the adsorption of arsenate by magnetite nanoparticles: kinetics and thermodynamic”***.

Iron ore tailings were utilized to make magnetite nanoparticles, which were then employed to filter out arsenate ions from the water. XRD, FTIR, TEM, and VSM investigations revealed that the created Sphere-shaped nanoparticles with magnetic properties have a diameter of 25–3 nm. The kinetics and stability like an

arsenate (As^{5+}) sorption by the magnetized nanoparticles were examined at $\text{pH}=5.5$ ($> \text{pHPZC}=3.8$). Due to $\text{CuH}_2\text{AsO}_4^+$ more volatility, oxidation reactions used to have a greater influence on the sorption of arsenate than other metal ions did. . Biphase first-order, pseudo-second-order, and Langmuir kinetic models were applied to simulate the binding of arsenate. The highest adsorption (9.72 mg/g) occurred at $\text{pH}=5.5$ with a starting concentration of arsenic of 13 mg/l , despite the fact that the sorption process was exergonic and increased as a function of temperature. Nevertheless, the creation of $\text{FeH}_2\text{AsO}_4^{2+}$ and $\text{AlH}_2\text{AsO}_4^{2+}$ groups in the context of Fe^{3+} and Al^{3+} cations at $\text{pH}=2.5$ reduced the As(V) sorption. The ability of the produced nanoparticles to be easily recovered and reused by NaOH solution suggests a viable adsorbent for As(V) removal.

TingWanget al. (2018) has published a journal titled ***“Adsorption of agricultural wastewater contaminated with antibiotics, pesticides and toxic metals by functionalized magnetic nanoparticles”***.

Due to their distinct physical and chemical characteristics, functionalized nanoparticles have found extensive usage in methods to remediate environmental concerns. Because different particular functional groups bind the surfaces of magnetic particles, which can effectively remove pollutants, MNPs have remarkable effectiveness towards the removal of pesticides, antibiotics, as well as harmful metals in agricultural wastewater. The purpose of the work presented here is to (i) summarize the literature on the sorption of pesticides, antibiotics, and toxic metals; (ii) discuss the sorption process and influence factors; and (iii) highlight future research needs and obstacles on the adsorption of agricultural wastewaters by MNPs. Greater efforts must be made to finance the relevant costs in order to make MNPs feasible for large-scale implementation.

Ata UrRehmanet al. (2021) has published a journal titled ***“Toxicity of heavy metals in plants and animals and their uptake by magnetic iron oxide nanoparticles”***.

Heavy metals are present in the environment naturally, but because of human activity and some natural processes, they have gotten into the air, the water, and the soil and have grown to be a significant worldwide problem. Most of them build up in the body for extended periods of time after entering it, causing a variety of

issues. For instance, they can damage important plant parts like bases, leaflets, and cellular constituents in addition to impeding vital biochemical activities like photosynthesis and mineral absorption. Similar to how they may harm an animal's essential bodily organs like the kidney and liver, they can also lead to deadly illnesses like cancer. The dose, length of exposure, and concentration of these heavy metals all have a significant role in the illnesses they cause. Because of the toxicity, bioaccumulation, and non-biodegradability of heavy metals, this issue has become quite serious. Various sources have exposed living organisms to these heavy metals, but drinking water is one of the main ones. To eliminate these metals from water, a lot of work has been done. However, the traditional methods have a number of shortcomings, such as being uneconomical and having an effect on the environment. Numerous benefits include regeneration, surface alteration, economic effectiveness, ease of usage, and environmental friendliness.

Fahad Saleem Ahmed Khan et al. (2020) has published scientific article titled ***"Magnetic nano adsorbents potential route for heavy metals removal—a review"***

The dumping of local waste and the fast expansion of the heavy metal-based businesses have had serious negative effects on the ecosystem. Reverse osmosis and ion exchange methods were commonly used in the past to remove heavy metals, but these processes have significant drawbacks. As a result, considerable effort has gone into creating better procedures, particularly for the removal of heavy metals. The use of nanotechnology offers the chance to improve already used methods in order to solve issues more successfully. Numerous novel materials, including magnetic nanoparticles, have been discovered as a result of advancements in nanotechnology. These nanoparticles have good qualities such as a high surface-to-volume ratio, less toxicity, and simple separation. Additionally, as compared to other conventional adsorbents, magnetic nanoparticles may be retrieved after adsorption quickly and effectively. The effectiveness of heavy metal removal from aqueous solutions using magnetic nano adsorbent is the major focus of this review. Additionally, a thorough examination of the methods used to create, characterize, and modify magnetic nanoparticles is provided in a methodical manner. Additionally, the potential benefits and drawbacks of utilizing magnetic nanoparticles as an adsorbent to remove heavy metals are highlighted.

Yuzhe Shiet *al.* (2020) has published scientific article titled ***“Synthesis of proanthocyanidins-functionalized Fe₃O₄ magnetic nanoparticles with high solubility for removal of heavy-metal ions”***.

A hydrothermal method was used to create large-scale hydrophilic proanthocyanidin-functionalized Fe₃O₄ nanoparticles. FTIR, SEM, TEM, and XRD is to employed the produced Fe₃O₄ nanocomposites (XRD). The findings showed that the Nanoparticles possessed an outside proanthocyanidin cap and a cubic spiral structure. Fe₃O₄ nanoparticles that have been functionalized with proanthocyanidins can also be used to separate particles containing metal ions from groundwater. The main findings suggested that the Fe₃O₄ nanoparticles functionalized with proanthocyanidins have a high rate of removal for Cu²⁺, Cd²⁺, and Pb²⁺ in water.

Noraziah Abu Yazidet *al.* (2019) has published a journal titled ***“Co-precipitation synthesis of magnetic nanoparticles for efficient removal of heavy metal from synthetic wastewater”***.

Magnetic nanoparticles (MNP) have emerged as a valuable tool or catalyst in a variety of commercial applications. MNP is smaller than bigger particles and as a result has a larger surface area, which renders it more responsive to particular chemicals. The main emphasis of this work is the application of MNP to the environmental problem associated with the heavy metal discharge from industrial effluents. In order to trap toxic metal ions in a complex that can be ejected by a magnetic drive, this study's goal is to synthesize MNP with a lower size. Varied variables, including temperatures (25°C, 50°C, 70°C) and bases (potassium hydroxide (KOH), sodium hydroxide (NaOH), and ammonium hydroxide (NH₄OH), added to a mixture containing iron(II) and iron(III), were used to synthesis the MNP in order to produce different sizes and morphologies. Atomic Adsorption Spectroscopy was used to assess the effectiveness of MNP removal on a solution containing Cr, Ni, and Cu before the results were examined (AAS). TEM, FTIR, and X-ray Diffraction were used to characterize the MNP (XRD). Based on the findings, it is determined that MNP produced with NH₄OH at 70°C is responsible for 90% of the Cr removal. The MNP has the shortest size (9 nm), which allows it to absorb more Cr ions. As a result, the strong base produces MNP with the

lowest dispersion, while the addition of surfactant results in less heavy metal adsorption but greater oxidation resistance. This research has shed light on the viability of using the MNP as a metal ion removal option.

Jin Zhanget al. (2020) has published a journal titled ***“Adsorption Properties of Magnetic Magnetite Nanoparticle for Coexistent Cr(VI) and Cu(II) in Mixed Solution”***.

Batch experiments were used to investigate the adsorption characteristics of coexistent Cr(VI) and Cu(II) in mixture solution on magnetite (Fe₃O₄) nanoparticles. A number of variables, including pH, adsorbent dosage, temperature, starting metal ion concentration, and coexisting ions in water, were studied for their effects. Adsorption isotherms and kinetics were both investigated at the same time. In order to better understand the process of adsorption for Cr(VI) and Cu(II), researchers looked at the surface characteristics of Fe₃O₄, the concentration of ions, and the effects of pH and zeta potential. The findings show that pH affects the adsorption of Cr(VI) and Cu(II), with 4.0 being the ideal pH for these adsorptions. With an increase in adsorbent dosage, adsorption efficiency improved. The temperature under the experimental setup had no discernible impact. In a mixture solution of 80 mg/dm³ Cr(VI) and Cu(II), respectively, the highest adsorption capacity for Cr(VI) and Cu(II) was achieved at 8.67 mg/g and 18.61 mg/g Fe₃O₄ concentration. Whereas other coexisting cations and anions had a few impact on the adsorption for Cr(VI), they had no effect on the adsorption for Cr(VI) and Cu(II). The Langmuir adsorption equation & the pseudo-second-order model provided a good match for the adsorption data for Cr(VI) and Cu(II). Nano-Fe₃O₄ was a versatile material that had strong adsorption performance for coexisting Cr(VI) and Cu(II) and was simple to separate and recover under magnetic field.

Mohamed A. Tahoonet al. (2020) has published a journal titled ***“Effective Heavy Metals Removal from Water Using Nanomaterials: A Review”***.

High harm to the environment is posed by the release of heavy metals such as Zn, Ni, Pb, Cu, Cr, Cd in water over the allowed limits. Better clean-up methods have recently been made available employing nanotechnology and nanomaterials. The focus was on newly created, cost-effective nanomaterials for use in water and

wastewater clean-up, including zeolite, carbonate, polymer-based, chitin, ferrous, metal oxide, bimetallic, and metallic materials. This review concentrated on the synthesis of different nano adsorbent materials and their capacity to remove various hazardous ions. Also studied was the impact of many experimental Physiochemical variables on the adsorption of heavy metals, including ionic strength, starting ionic strength, temperature, contact duration, adsorbent dosage, and pH.

K. Anbalagan et al. (2021) has published scientific article titled ***“Removal of heavy metal ions from industrial wastewater using magnetic nanoparticles”***.

By the co-precipitation approach, maghemite ($\gamma\text{-Fe}_2\text{O}_3$) and MNP (Fe_3O_4), were created in this study, and they were characterized using XRD, SEM, EDS, and FTIR Spectroscopy. These particles, which ranged in size from 20 to 100 nm, were utilised as nano adsorbents to filter out toxic metals like copper and chromium from synthetic wastewater. Adsorption is dependent on factors including pH (2.5 & 6.5), contact duration (60 and 80 minutes), and doses at room temperature, which were shown to have a 92% removal effectiveness for chromium and copper. These particles demonstrated astounding metal removal effectiveness. Additionally, these particles were renewed for reuse; for the desorption of copper and chromium metals, respectively, HCL and NaOH effluent were used. The observed data clearly show that magnetic nanoparticles have improved pollution removal. The separation factor (r), which ranges from 0 to 1, shows that the Langmuir isotherm favors both the ions of copper and chromium.

CHAPTER-3 AIM AND SCOPE

The purpose of this thesis is to create a unique nanocomposite for the removal of zinc from waste water that has improved capacity and is easy to use, cost effective, and friendly to the environment. The goals of this study were to

- (1) generate metal oxide nanocomposites(Fe_2O_3)
- (2) apply the obtained nanocomposites(Fe_2O_3) to the adsorption of Zn^{2+} in water
- (3) explore the process of adsorption of these synthetically produced materials.

3.1 DEVELOPMENT OF NANOCOMPOSITE MATERIALS

The manufacture of calcium oxide that had been impregnated with Fe_2O_3 was the first step in this investigation. Next, the material was characterized. Nanoparticles made of iron oxide that have been synthesised and are magnetic. These nanoparticles are designed to bind heavy metal ions and have coordinating or chelating groups on their surfaces. Magnetic measurements will be used to examine the nano composites in order to assess the separation efficacy of the magnet, which is necessary in order to determine whether or not this removal system will be successful. Following this step, the nanocomposites will be utilised in adsorption tests with the purpose of removing Zn^{2+} . If the nanocomposites were put directly to waste water, they would be more effective. This is especially true when one considers the fact that waste water is typically held in containers in order to prevent the sewage system from becoming overloaded. During the process of Zn^{2+} removal, this would prevent the problems that are caused by a high concentration of suspended matter. The inquiry makes use of calcium oxide, which is a type of metal oxide. Calcium oxide, often known as CaO , has a large capacity and performs exceptionally well as an adsorbent for a wide variety of hazardous compounds. As a result, the purpose of this study is to design a method that is efficient, uncomplicated, and sensitive for extracting zinc from aqueous solutions by utilising nanocomposite calcium oxide (CaO), as well as to discover the circumstances under which this method is most effective. An examination of the adsorbent's surface shape, as well as its chemical and physical properties, was carried out with the assistance of XRD, FTIR, and SEM/EDX.

3.2 ADSORPTION EXPERIMENTS AND STUDIES

The removal of heavy metals can be accomplished using a variety of processes, including chemical oxidation and advanced oxidation, chemical precipitation, chemical coagulation, chemical stabilization, and ion exchange. Nanotechnology has been exploited in recent efforts that have been made to purge effluent of heavy metal ions. Adsorption and membrane separation are the two methods that are used the most frequently. For the purpose of effectively removing heavy metals, numerous nanomaterials, including those based on carbon, metal oxides, zero-valent nanoparticles, and nanocomposites, have been produced. The high removal capacity and selectivity of these metal oxide nanoparticles are two of the many reasons why they are advantageous. The effect of a number of parameters, including the pH of the solution, the initial concentration of chromium, and the amount of adsorbent used, was investigated. After each test, the CaO/Fe₂O₃ nanocomposite adsorbent was removed from the aqueous solution, and the concentration of Zn²⁺ ions in the solution was measured. We performed the calculations necessary to determine the adsorption capacity of Zn²⁺ ion as well as its removal efficiency from aqueous solution. The efficacy of the process can be mostly attributed to the type of adsorbent that is used, which is dependent on the concept that the process will result in a net buildup of metal at the surface of the solid phase (adsorbent). The process of adsorption can be broken down into three distinct phases: the transport of the adsorbate from the liquid phase to the solid adsorbent surface, the adsorption of the analytes onto the solid adsorbent surface, and the transit of the adsorbate within the adsorbent particle. As a result, this article discusses the processes of adsorption as an efficient way for eliminating hazardous metal pollutants from wastewater.

CHAPTER-4 MATERIALS AND METHODOLOGY

4.1 MATERIALS

The List of materials employed to synthesize $\text{CaO-Fe}_2\text{O}_3$ are listed below:

4.1.1 Chemicals required

- ❖ Hydrochloric Acid (HCl)
- ❖ Ferric Chloride (FeCl_3)
- ❖ Ferric Sulphate ($\text{Fe}_2(\text{SO}_4)_3$)
- ❖ Calcium Oxide(CaO)
- ❖ Sodium hydroxide (NaOH)
- ❖ Distilled Water
- ❖ Ammonia solution (NH_3)

4.1.2 Equipments required

- ❖ Muffle Furnace
- ❖ Hot Air Oven
- ❖ Hot Plate Magnetic Stirrer
- ❖ Rotary Shaker

4.2 METHODOLOGY

Over the course of the past few years, nano-structured metal oxide has established itself as the best adsorption candidate for the treatment of wastewater. Nanoparticles made of metal oxide have a number of properties in common with other nanomaterials, including large pore volumes and surface areas, high adsorption capacities, pores of a good size, and sites that can be functionalized. Iron has the position as the fourth most abundant element found in the crust of the Earth. These nanomaterials have been the focus of a significant amount of research as a direct result of the ease and accessibility with which iron oxides may be produced. They outperform other adsorbing substances in terms of their capacity for adsorption, their affinity for the material being adsorbed, and their rate of adsorption. Iron nanoparticles are frequently used in the process of removing hazardous metals from water and wastewater. Chemical precipitation was used to create the nanocomposite of CaO and Fe_2O_3 , which was then characterized. The Fig.4.1 shows the process involved in the treatment.

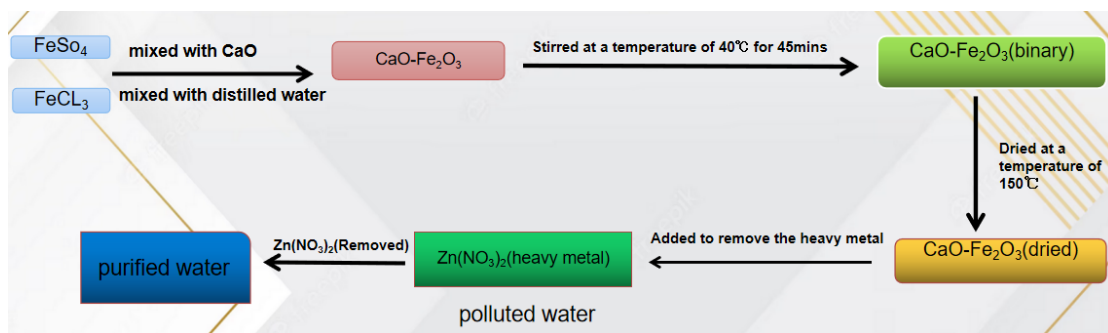


Fig.4.1 Methodology

4.2.1 $\text{CaO/Fe}_2\text{O}_3$ nanocomposite synthesis

It has been determined how to make a solution with a molecular ratio of 1:2 that includes both ferric chloride and ferric sulphate. Following this step, roughly 1 g of prepared CaO was added to the mixture, and it was agitated for a total of 20 minutes until the iron ions formed bonds with the CaO particles (Fig 4.2). After bringing the temperature of the solution up to between 80 and 90 degrees Celsius,

thirty ml of NH_4OH were added to the combination in a drop-by-drop fashion, and then the mixture was permitted to sit at the designated temperature for half an hour (Fig 4.3). The manufactured magnetic composite has been isolated from the solution by using filter paper to make the separation (Fig 4.4). After that, the composite that had been created was allowed to dry at a temperature of 150 degrees Celsius for two hours before it was put to use as an adsorbent to get rid of Zn^{2+} ions.

4.2.2 Stock solution of Zn^{2+}

Deionized water that had been distilled twice was used in the preparation of the Zn^{2+} ion stock solution. By dissolving 29g of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in double-distilled water, a stock solution with a concentration of 100 mg/L was produced. Other stock solutions with concentrations of 20, 40, 60, 80, and 100 mg/L were created using this stock solution as a starting point and then diluted with double-distilled water. The prepared stock solution is shown in the figure 4.5

4.2.3 Adsorption experiments and studies

During the adsorption experiments, flasks containing 250ml and 100ml of a solution containing Zn^{2+} ions were loaded with $\text{CaO}/\text{Fe}_2\text{O}_3$ nanocomposite as an adsorbent. The effect of a number of parameters, including solution pH (which was explored at 2, 3, 4, 5, 6, and 7), starting chromium content (which was researched at 20, 40, 60, 80, and 100 mg/L), and adsorbent dosage (which was studied at 0.3, 0.4, 0.5, 0.6, and 0.7 g), was investigated and it is shown in the figure 4.6. Following each experiment, the $\text{CaO}/\text{Fe}_2\text{O}_3$ nanocomposite adsorbent was removed from the aqueous solution, and the amount of Zn^{2+} ion that remained in the aqueous solution was calculated. The following equation for mass balance was used to determine the adsorption capacity of the Zn^{2+} ion in aqueous solution as well as the removal efficiency of that ion.

$$\% \text{ Adsorption} = \frac{C_i - C_e}{C_i} \times 100 \dots\dots\dots(1)$$

where C_i is the initial Zn^{2+} concentration in $mmol\ L^{-1}$; C_e is Zn^{2+} final concentration in $mmol\ L^{-1}$.

The amount of Zn^{2+} adsorbed ($mg\ g^{-1}$) at time t was calculated by using the following equation:

$$q_t = \frac{(C_0 - C_t)V}{m} \dots\dots\dots(2)$$

where C_0 and C_t are Zn^{2+} concentrations ($mg\ L^{-1}$) initially and at a given time t , V is the volume and $m(g)$ is the mass of the adsorbent.

4.2.4 Adsorption isotherms

The adsorption isotherm between the the adsorbate and adsorbent is typically used to characterize adsorption. In general, an adsorption isotherm is a useful method for describing the phenomenon regulating the retention (or release) or mobility of a substance from aqueous media to solid-phase at a constant temperature and pH. The experimental data are utilised to construct adsorption isotherms. Langmuir and Freundlich are the two primary models used to characterize the adsorption process on solid surfaces. These models can be applied to a broad spectrum of adsorbate concentrations to provide relevant and essential information for design and scaling. The Langmuir model believes that the adsorption of heavy metals acts as a mono-layer on a homogeneous surface through the equilibrium distribution of metal ions within the solid and liquid phases, with the non-linear form represented by equation.

$$q_e = \frac{q_{max} k_L C_e}{1 + k_L C_e} \dots\dots\dots(3)$$

q_{max} is the maximal adsorption capacity that corresponds to site saturation ($mg\ g^{-1}$), C_e is the equilibrium metal concentration ($mg\ L^{-1}$), and k_L is the Langmuir constant ($L\ mg^{-1}$). The Langmuir isotherm implies that metal ions are chemically adsorbed, in which each site can contain only one adsorbate, all sites are energetically equivalent, and ion interactions do not occur.

Equation can be rearranged to the following linear form to be used for the linearization of the experimental data by plotting C_e/q_e vs C_e :

$$\frac{C_e}{q_e} = \frac{1}{k_L q_{\max}} + \frac{C_e}{q_{\max}} \dots \dots \dots (4)$$

The Freundlich model, which is an empirical model, assumes the presence of preferential sites in the adsorbent and can be expressed in the non-linear form of equation

$$q_f = k_f C_f^{1/n} \dots \dots \dots (5)$$

where q_f is defined as the amount of adsorbate adsorbed in mg g^{-1} and C_f is the concentration of adsorbate at equilibrium.

The Freundlich equation can be linearized by taking the logarithm of both sides of the equation where k_f (mg g^{-1}) is the Freundlich constant related to the adsorption capacity and $1/n$ is the heterogeneity factor:

$$\log q_f = \log k_f + \frac{1}{n} \log C_f \dots \dots \dots (6)$$

4.2.5 Adsorption kinetic equations

Kinetic models are used to analyse and interpret the process adsorption mechanisms derived from batch elimination of metal ions experimental data. Kinetics, often known as reaction rate, is defined as the movement or change in concentration of a reactant or product over time. Adsorption kinetics dynamics explain the pace at which the adsorbate is adsorbed onto the adsorbent, controlling the time at the particle-metal interface. Two kinetic models were used

to explore the mechanism of sorption: (i) a pseudo-first-order kinetic model and (ii) a pseudo-second-order kinetic model. Lagergren's (pseudo-first-order) model is a model that is expressed as follows and is dependent on the amount of metal ion adsorption increased to the first power:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \dots\dots\dots(7)$$

When applying the initial conditions for the definite integration $q = 0$ at $t = 0$ and $q=q_t$ at $t=t$.

Equation is linearized to :

$$\log (q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303} \right) t \dots\dots\dots(8)$$

where k_1 is the pseudo-first-order rate constant (min^{-1}) of adsorption, while q_e and q_t are the adsorption capacities of Zn^{2+} (mg g^{-1}) at equilibrium and at time $t(\text{min})$. The pseudo-second-order model is a model that depends upon the amount of metal ions on the surface of the adsorbent at time t and that adsorbed at equilibrium adsorption raised to the second power is expressed as follows:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \dots\dots\dots(9)$$

When applying the initial conditions for the definite integration $q = 0$ at $t = 0$ and $q=q_t$ at $t=t$, equation is linearized to

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e} \right) t \dots\dots\dots(10)$$

where k^2 is the pseudo-second-order rate constant of adsorption ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$). The mechanism for metal adsorption onto adsorbents consists of four major steps:

- (a) Zn^{2+} ion transfer and migration from solution to the boundary layer at the adsorbent surface (bulk diffusion)
- (b) metal ion (Zn^{2+}) migration from the boundary layer to the adsorbent surface via film diffusion
- (c) transporting metal ions from the surface to the particles' interior pores
- (d) Zn^{2+} ion adsorption employing active sites on the adsorbent surface induced by chemical processes such as ion exchange.



Fig.4.2 Impregnation



Fig.4.3 Magnetic solution



**Fig.4.4 $\text{CaO-Fe}_2\text{O}_3$
nanocomposite**



Fig.4.5 Stock solution



Fig.4.6 Optimization samples

CHAPTER-5 RESULTS AND DISCUSSION

The overarching goal of this section of the thesis was to locate and make use of nanomaterials that had just been produced or were already on the market. When it comes to the removal of heavy metals from aquatic systems, the nanoparticles were put up against some commonly used adsorbents for comparison. The dissolvability of both commercially available metal oxide and nanoparticles manufactured in the lab was investigated. The degree to which metal oxide nanoparticles dissolve can be important information for toxicology researchers to have. The effects of researching various characteristics on the adsorption of heavy metals such as Zn^{2+} are examples of systems that were examined, and the results are presented below. Some of these parameters include contact time, initial ion concentrations, dosage, and solution pH.

5.1 THE MORPHOLOGY OF THE SURFACES OF NANOMATERIALS

There are currently a wide variety of materials that are utilised for the removal of heavy metals from aqueous systems, in addition to increasing qualities that can either rehabilitate or eliminate heavy metals. Despite the fact that these materials have been demonstrated to be useful, they do have some drawbacks. When considering the implications of commercially available and lab-made nanoparticles for use in biological applications, the surface formulation of the nanoparticles is an important consideration. It is possible for nanoparticles to produce cytotoxicity in human or animal cells when they interact with other cell types.

5.1.1 FESEM/EDX

The results of an investigation employing FESEM/EDX on the surface morphology and elemental distribution of a $\text{CaO-Fe}_2\text{O}_3$ nanocomposite are shown below in the Fig 5.1, 5.2 and 5.3 and Table 5.1.

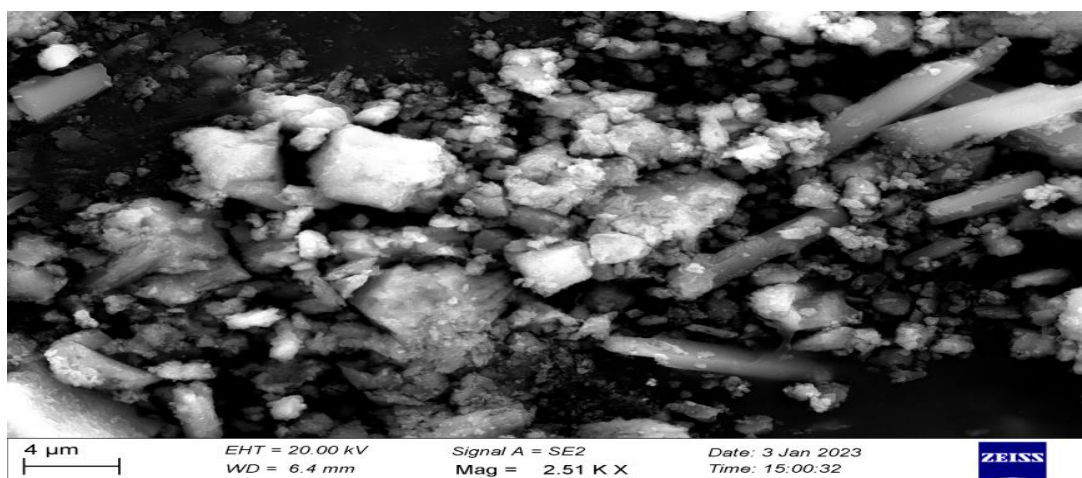
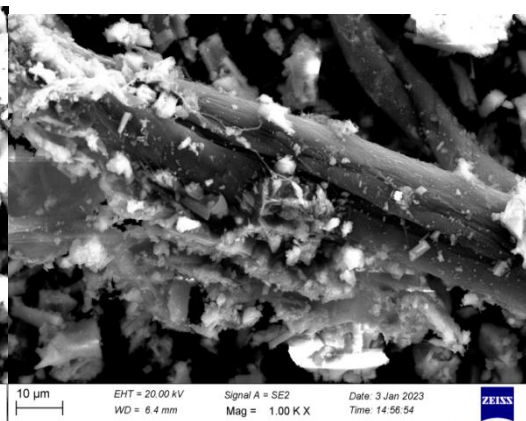
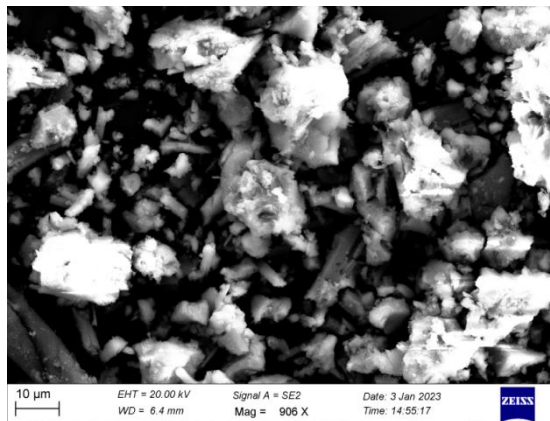
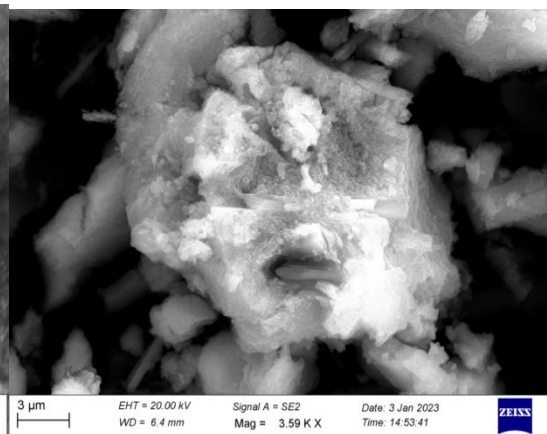
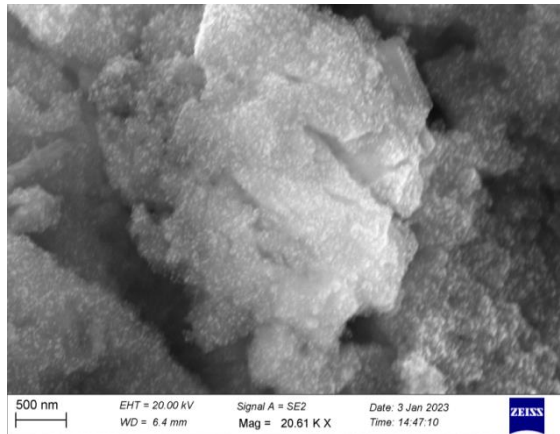
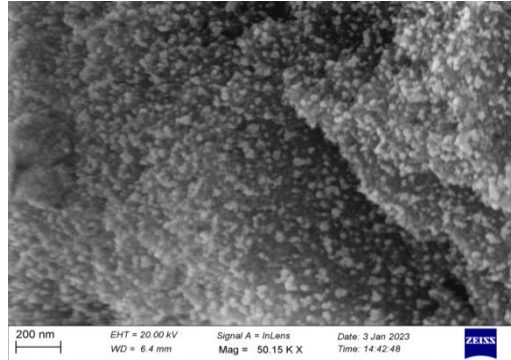
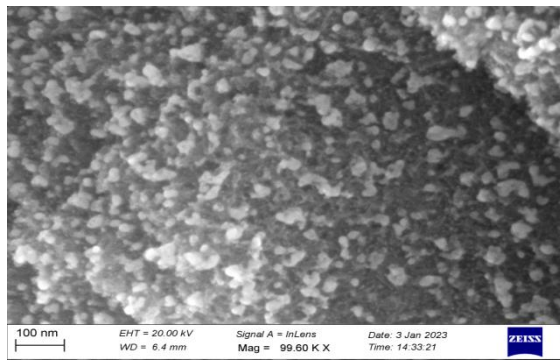


Fig.5.1 SEM images of $\text{CaO-Fe}_2\text{O}_3$

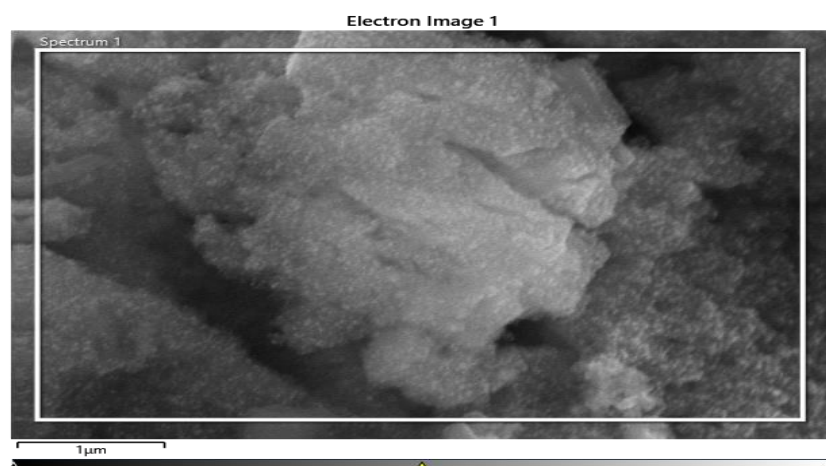


Fig.5.2 EDX of CaO-Fe₂O₃

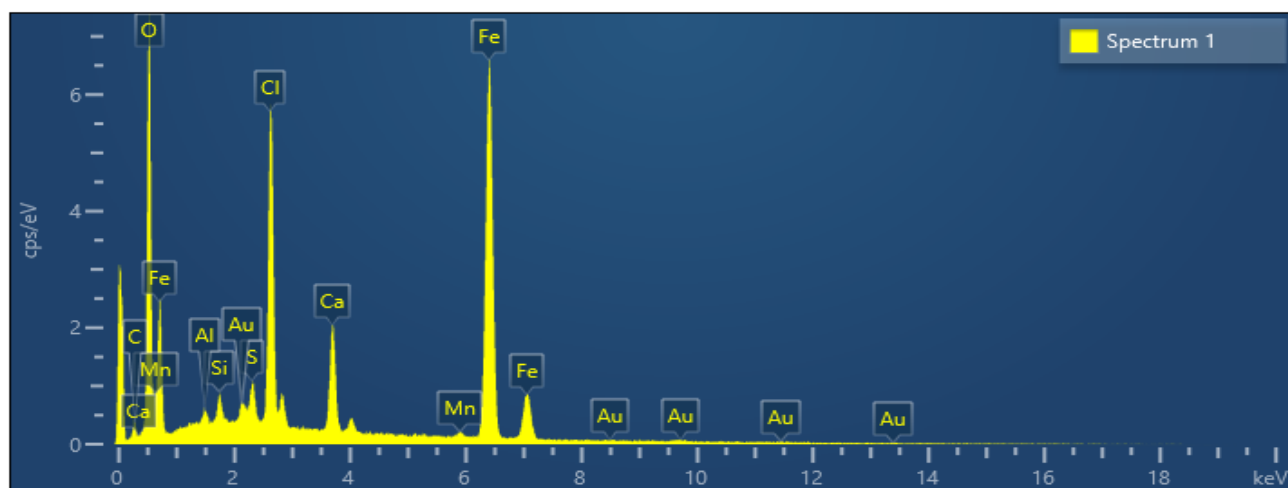


Fig.5.3 EDX graph of CaO-Fe₂O₃

Table 5.1 The elemental composition of CaO/Fe₂O₃ nanocomposite

Spectrum 1				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	6.36	0.81	13.73
O	K series	33.84	0.39	54.80
Al	K series	0.46	0.05	0.44
Si	K series	0.74	0.05	0.69

S	K series	1.14	0.05	0.92
Cl	K series	9.67	0.12	7.07
Ca	K series	4.27	0.08	2.76
Mn	K series	0.44	0.06	0.21
Fe	K series	41.30	0.42	19.16
Au	M series	1.77	0.18	0.23
Total		100.00		100.00

In the process of adsorbing Zn^{2+} ions, the surface hardness and particle size of the $\text{CaO-Fe}_2\text{O}_3$ nanocomposite can vary, which is something that is both highly significant and effective. After Zn^{2+} adsorption, the surface of the $\text{CaO-Fe}_2\text{O}_3$ nanocomposite has altered. This may be attributed to the substitution of the ions on the nanocomposite surface with Zn^{2+} ions, which caused the surface to undergo the change. Investigations using FESEM and EDX revealed the presence of Zn^{2+} ions in the structure of the magnetic composite material. The findings of these investigations also showed that the surface of the substance contained carbon, oxygen, iron, and calcium in an even distribution. The formation of magnetic nanoparticles made of Fe_2O_3 could be responsible for the presence of iron on the surface of the adsorbent.

5.1.2 FTIR analysis

The following image (Fig 5.4) presents the findings of an FTIR analysis performed on $\text{CaO-Fe}_2\text{O}_3$ nanocomposite after the adsorption of Zn^{2+} ions. A prominent and wide peak was detected in the wavelength range of 1792 and 1838 cm^{-1} for CaO that was generated from eggshell. This peak was an indication of vibration of functional groups of N-H and O-H that are present in the structure of CaO. Additionally, peaks in the wavelength ranges of 1332, 1509, 1553, and 2419 cm^{-1} were identified, each of which indicated that functional group vibrations of C = O and C-O-O were present, respectively. The presence of Ca-O in the structure of calcium oxide is the cause of the minute peak that can be found at 974 cm^{-1} .

During the process of creating the CaO-Fe₂O₃ nanocomposite, some peaks in the CaO structure were also altered. These variations could be induced by the formation of Fe₂O₃ nanoparticles in the CaO structure. In addition, the steep peak that was seen in the region of 3887 cm⁻¹ could be attributable to Fe-O vibrations that were present in the composite structure. Recent research has revealed that interactions between functional groups and Zn²⁺ ions are responsible for the changes that took place in the CaO structure as a result of using CaO-Fe₂O₃ nanocomposite as the adsorbent. These interactions were observed in the CaO-Fe₂O₃ nanocomposite.

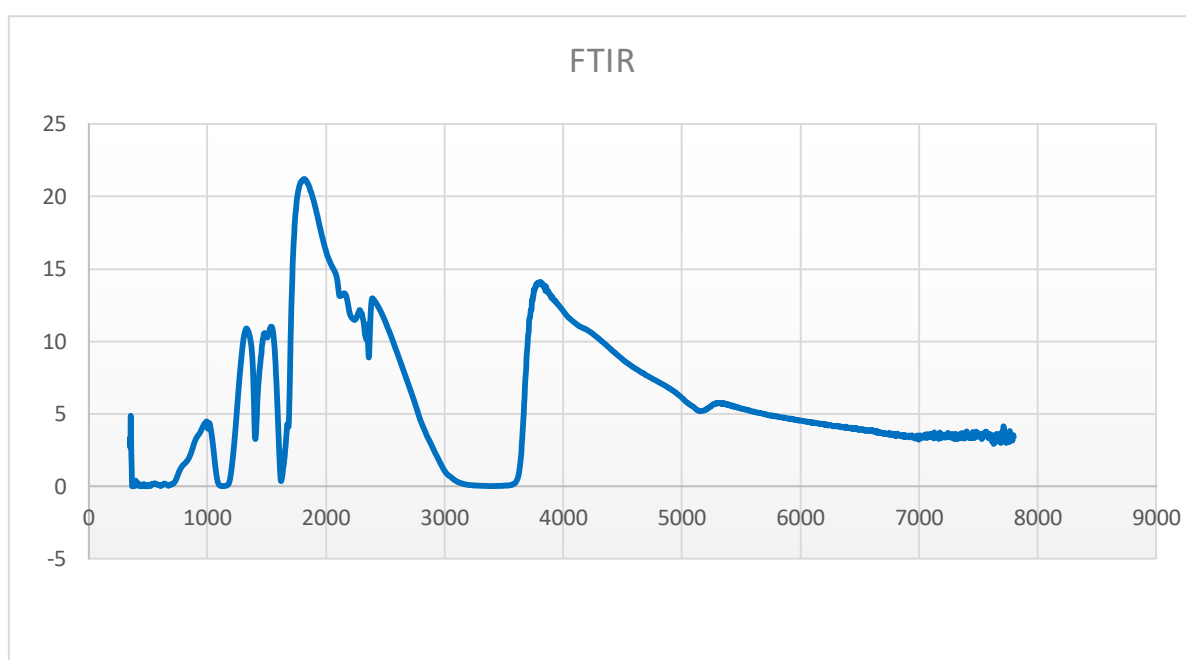


Fig.5.4 Graphical representation of FTIR analysis

5.1.3 XRD analysis

The results of an XRD examination that was conducted to determine the crystalline phases of CaO and a composite of CaO and Fe₂O₃ are shown in Fig 5.5 for your perusal. According to the data, the fact that CaO-Fe₂O₃ exhibits a number of peaks at various angles indicates that both of the aforementioned substances have a crystalline structure. In the CaO-Fe₂O₃ composite structure, both the range and intensity of the peaks that were present in the CaO structure were significantly altered. Also, multiple peaks at 2 of 11.64°, 20.77°, 29.19°, 38.19°, 40.19°, 43.19°, 45.19°, 47.19°, 49.19°, 51.19°, 53.19°, 55.19°, 57.19°, 59.19°, 61.19°, 63.19°, 65.19°, 67.19°, 69.19°, 71.19°, 73.19°, 75.19°, 77.19°, 79.19°, 81.19°, 83.19°, 85.19°, 87.19°, 89.19°, 91.19°, 93.19°, 95.19°, 97.19°, 99.19°, 101.19°, 103.19°, 105.19°, 107.19°, 109.19°, 111.19°, 113.19°, 115.19°, 117.19°, 119.19°, 121.19°, 123.19°, 125.19°, 127.19°, 129.19°, 131.19°, 133.19°, 135.19°, 137.19°, 139.19°, 141.19°, 143.19°, 145.19°, 147.19°, 149.19°, 151.19°, 153.19°, 155.19°, 157.19°, 159.19°, 161.19°, 163.19°, 165.19°, 167.19°, 169.19°, 171.19°, 173.19°, 175.19°, 177.19°, 179.19°, 181.19°, 183.19°, 185.19°, 187.19°, 189.19°, 191.19°, 193.19°, 195.19°, 197.19°, 199.19°, 201.19°, 203.19°, 205.19°, 207.19°, 209.19°, 211.19°, 213.19°, 215.19°, 217.19°, 219.19°, 221.19°, 223.19°, 225.19°, 227.19°, 229.19°, 231.19°, 233.19°, 235.19°, 237.19°, 239.19°, 241.19°, 243.19°, 245.19°, 247.19°, 249.19°, 251.19°, 253.19°, 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2729.19°, 2731.19°, 2733.19°, 2735.19°, 2737.19°, 2739.19°, 2741.19°, 2743.19°, 2

29.22°, 35.81°, and 62.63° are detected in the structure of CaO-Fe₂O₃ composite. These peaks can be attributed to the crystalline phases of (1000), (587), (641), (36), and (355), respectively, and demonstrate that Fe₂O₃ is present in the structure of CaO-Fe₂O₃ composite. As a consequence of this, these peaks demonstrate that Fe₂O₃ was manufactured in an appropriate manner.

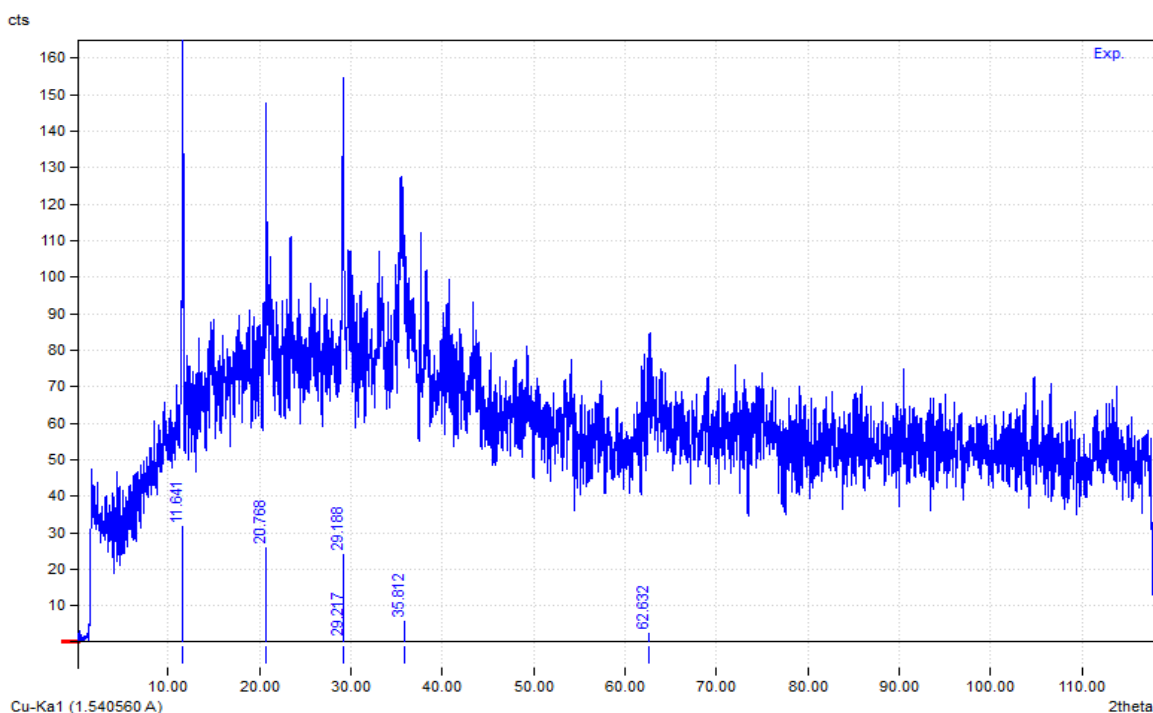


Fig.5.5 XRD image of CaO-Fe₂O₃

5.2 OPTIMIZATION OF ADSORPTION PARAMETERS

5.2.1 The influence of pH on the adsorption process

Adsorption of Heavy Metal Ions pH of the Solution The pH of the solution is very important in the adsorption of heavy metal ions. The influence of pH (in the range of 2-8) on the adsorption of Zn²⁺ by CaO-Fe₂O₃ nanocomposite is depicted here using a graph (Fig 5.6). The adsorption effectiveness of Zn²⁺ increased considerably from 52.56 percent to 97.32 percent when the pH was between 2 and 6. Due to the fact that Zn²⁺ and hydrogen ion (H⁺) both attempted to adsorb onto the active sites of the CaO-Fe₂O₃ nanocomposite at low pHs, the adsorption

efficiency was significantly reduced. Because free Zn^{2+} ions are present in the forms of ZnOH^{2+} , $\text{Zn}(\text{OH})_4^{4+}$, and $\text{Zn}_3(\text{OH})_4^{5+}$ for adsorption on the negative side of the $\text{CaO-Fe}_2\text{O}_3$ nanocomposite, it was discovered that the adsorption of Zn^{2+} at a pH of 3-5 is favourable. In addition, the adsorption efficiency and the slope of the plot both decreased for pH values that were larger than 6. This could be because of competition on the active sites of the $\text{CaO-Fe}_2\text{O}_3$ nanocomposite between the ion Zn^{2+} that results from precipitation and the hydroxyl ion that results from adsorption. As a consequence of this, a pH value of 6 was concluded to be optimal, and this value was utilised in subsequent research.

The influence that adsorbent dose has on the adsorption process The efficacy and capacity of adsorption of Zn^{2+} ion on $\text{CaO-Fe}_2\text{O}_3$ nanocomposite at adsorbent dosage range of 0.1-0.5 g/L are shown in the figure. The adsorbent dosage range was 0.1-0.5 g/L. The graph makes it abundantly evident that increasing the amount of adsorbent used improves the efficiency of the adsorption process. In addition, the adsorption equilibrium was found to be achieved at a dosage of 0.1 g/L for the adsorbent. At an adsorbent dosage of 0.1 g/L, it was found that the maximum adsorption efficiency could be achieved, which was 88.53%. At adsorbent dosages higher than 0.1 g/L, there was no detectable improvement in the effectiveness of the adsorption process. It is possible that an increase in the availability of active sites is the cause of an increase in the adsorption efficiency that occurs when the dosage of the adsorbent is raised. In addition, as a consequence of the saturation of available adsorption sites, the level of adsorption efficiency that was present at the point of equilibrium did not change. As the amount of adsorbent rose, the capacity for adsorption decreased. This result can be dictated by the interference and collision of adsorbent active sites at a higher adsorbent dosage, or it can be that the concentration of Zn^{2+} ion in solution is inadequate to establish bonds with accessible and active sites on the adsorbent surface of $\text{CaO-Fe}_2\text{O}_3$ nanocomposite. adsorbent active sites can interfere with each other and collide.

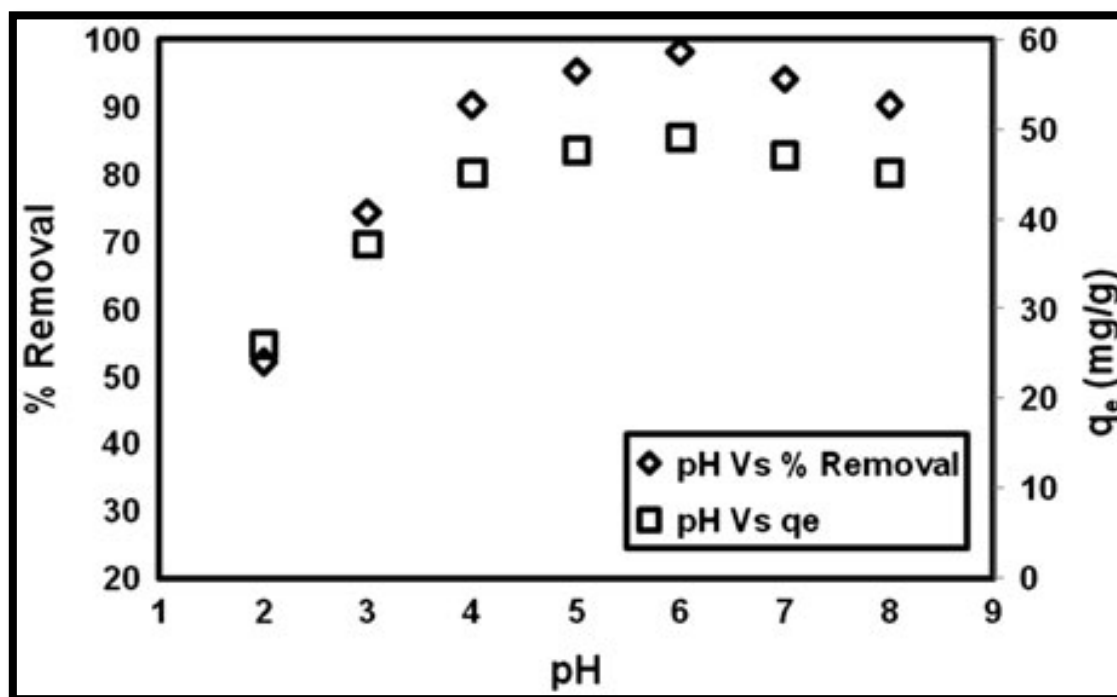


Fig.5.6 Effect of pH on CaO-Fe₂O₃ with Zn²⁺

5.2.2 The influence of the starting concentration of Zn²⁺ ions

We looked into how the beginning concentration of Zn²⁺ ions affected the amount of Zn²⁺ ions that could be adsorbed by a nanocomposite of CaO and Fe₂O₃. The graph (Fig 5.7) shows that the adsorption effectiveness improved as the initial concentration of Zn²⁺ ion became higher. This result might be explained by an increase in the initial concentration of Zn²⁺ ion, which provides the necessary driving forces for mass transfer between the liquid and solid phases. It has been discovered that the ratio of accessible active sites to Zn²⁺ ions in the aqueous phase is larger when the concentration of the Zn²⁺ ions is lower. When the concentration was increased, however, the active site became saturated, which caused the ratio to decrease. As a consequence of this, the efficiency of the adsorption process was 17.73% when the starting concentration of Zn²⁺ was 50 mg/L.

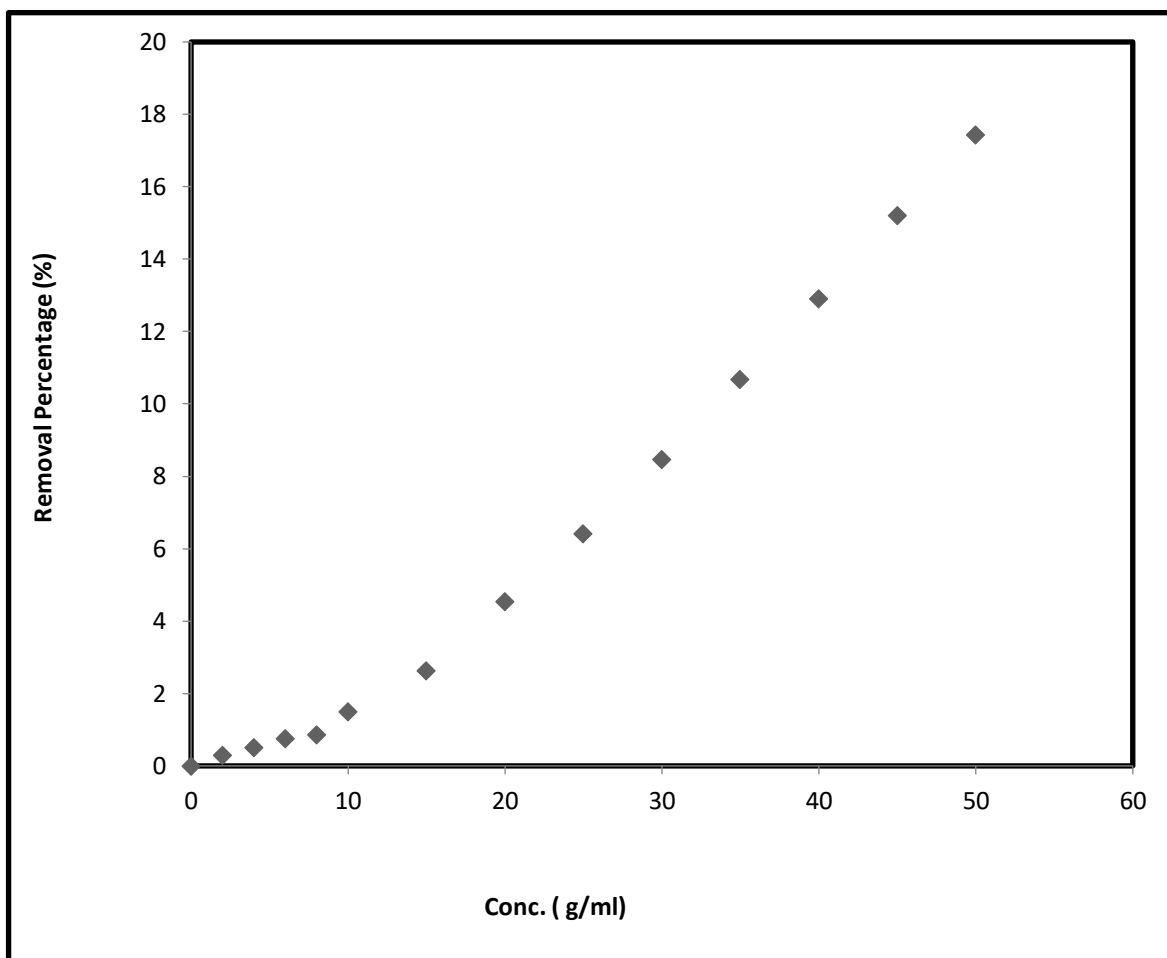


Fig.5.7 Effect of concentration on CaO-Fe₂O₃ with Zn²⁺

5.2.3 The influence of contact duration on the adsorption process

We looked at how the amount of time that Zn²⁺ ions were in contact with the CaO-Fe₂O₃ nanocomposite affected the adsorption performance of Zn²⁺ ions by the nanocomposite. As can be seen in the figure 5.8, increasing the contact duration from 10 to 180 minutes resulted in Zn²⁺ adsorption by CaO-Fe₂O₃ nanocomposite occurring rapidly. After achieving equilibrium at 180 minutes, the rate of adsorption began to gradually decline. There is a possibility that the greater accessibility of active sites in the CaO-Fe₂O₃ nanocomposite is the cause of the quicker adsorption at initial contact times. In addition, a drop in adsorption efficacy during the course of the contact time may be the result of saturation, which leads to a reduction in the number of active sites. In light of these data, it is reasonable to conclude that a contact time of one hundred and fifty minutes is adequate for Zn²⁺ ion adsorption under the present experimental conditions.

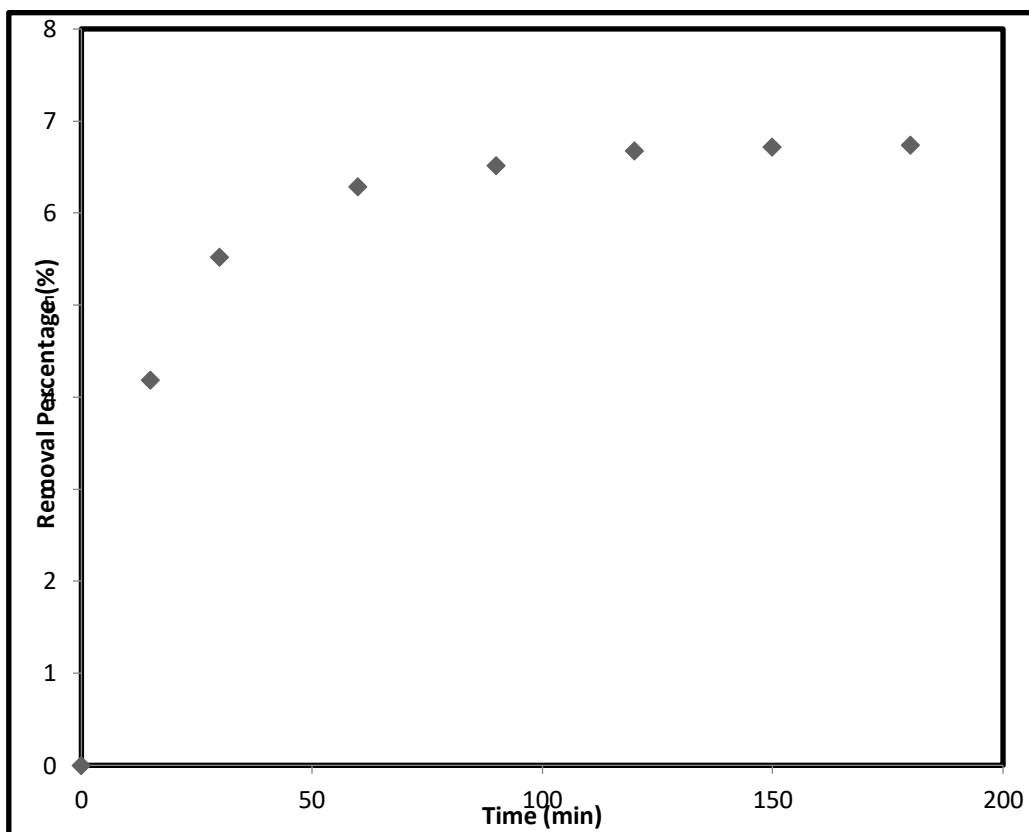


Fig5.8 Effect of contact time on CaO-Fe₂O₃ with Zn²⁺

5.2.4 The influence of temperature on the process of adsorption

The influence of temperature on the efficiency of Zn²⁺ adsorption by CaO-Fe₂O₃ nanocomposite from aqueous solution is depicted in the graph (Fig 5.9). According to the figure, an increase in the adsorption efficiency from 93.87% to 96.73% was achieved by lowering the temperature from 310 to 300 K. This result may be explained by an increase in the reaction that occurs between the Zn²⁺ ion and the active sites of the CaO-Fe₂O₃ nanocomposite. This observation provides further evidence that the adsorption of Zn²⁺ ions to the CaO-Fe₂O₃ nanocomposite generates heat. In addition, it was demonstrated that the efficacy of adsorption decreased below 320 K, which can be related to the instability of the Zn²⁺ ion and the formation of complexes at lower temperatures. The adsorption of Zn²⁺ on CaO-Fe₂O₃ nanocomposite at room temperature is the recommended method due to the favourable economics of this approach.

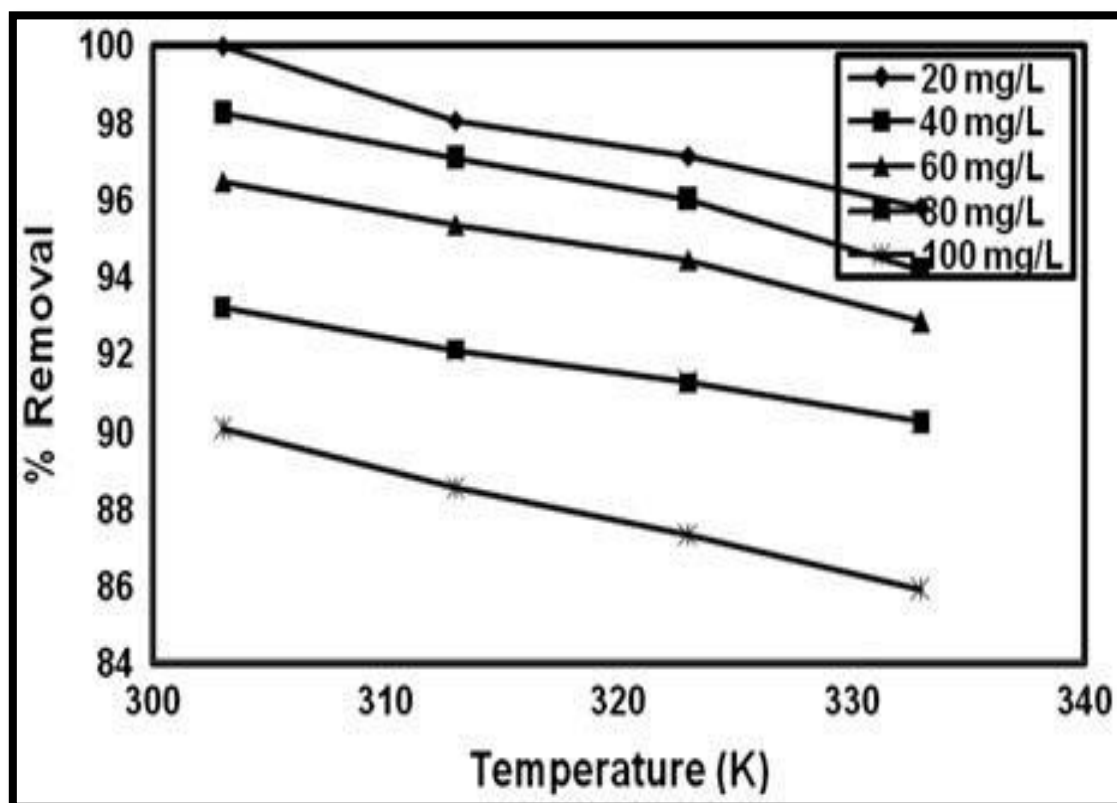


Fig.5.9 Effect of Temperature on CaO-Fe₂O₃ with Zn²⁺

5.3 THE ISOTHERMS OF ADSORPTION

5.3.1 The Isotherm of Langmuir

The Langmuir isotherm model for Zn²⁺ adsorption on CaO-Fe₂O₃ nanocomposite was utilized, and the findings are shown in Fig 5.10 and Table 5.2. The concentrations used ranged from 10 to 80 mg/L. The value of the regression coefficient (R^2) for Zn²⁺ was calculated to be 0.961. In addition, the KL value was determined to be in the range of 0.092, which, given the conditions described earlier, is appropriate. In addition, the CaO-Fe₂O₃ nanocomposite exhibited a maximum adsorption capacity of 17.857 mol g⁻¹. As a direct consequence of this, the adsorption of Zn²⁺ ion using CaO-Fe₂O₃ nanocomposite adheres to the Langmuir isotherm model and takes place on the homogeneous sites and monolayer surfaces of the CaO-Fe₂O₃ nanocomposite adsorbent.

Table 5.2 Langmuir adsorption isotherm parameters

Adsorbent	$K_L \times 10^{-4} (\text{L mol}^{-1})$	$q_m \times 10^6 (\text{mol g}^{-1})$	R^2
CaO -Fe ₂ O ₃	0.092	17.857	0.961

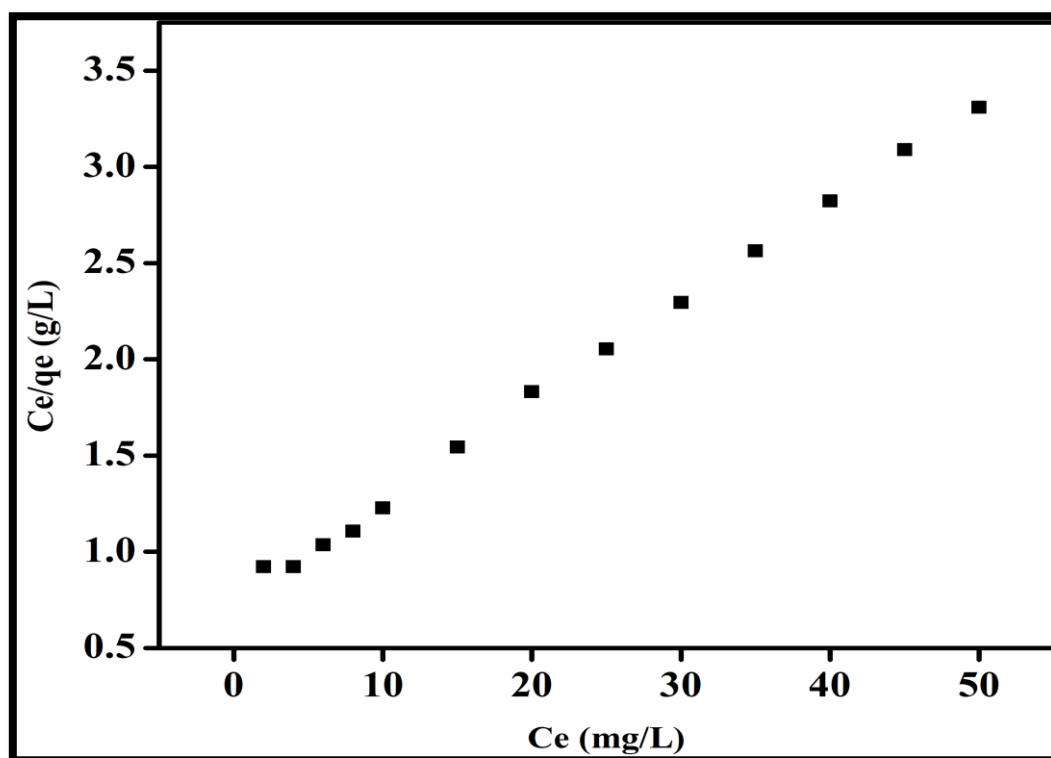


Fig.5.10 Langmuir Isotherm graph on CaO-Fe₂O₃ with Zn²⁺

5.3.2 Freundlich Isotherm

The results of the Freundlich isotherm model as well as the isotherm model of Zn²⁺ ion adsorption onto CaO-Fe₂O₃ nanocomposite are provided below in the Fig 5.11 and Table 5.3. In Zn²⁺ ion adsorption by CaO-Fe₂O₃ nanocomposite, the constant value of n was estimated to be 1.48, which indicates low adsorbent surface heterogeneity. In a similar manner, it was found that the determination coefficient (R^2) of the Zn²⁺ ion was 0.946.

Table 5.3 Freundlich adsorption isotherm parameters

Adsorbent	$K_F \times 10^2$ ((mol g ⁻¹) (mol L ⁻¹) ^{-1/n})	N	R ²
CaO-Fe ₂ O ₃	1.48	1.557	0.946

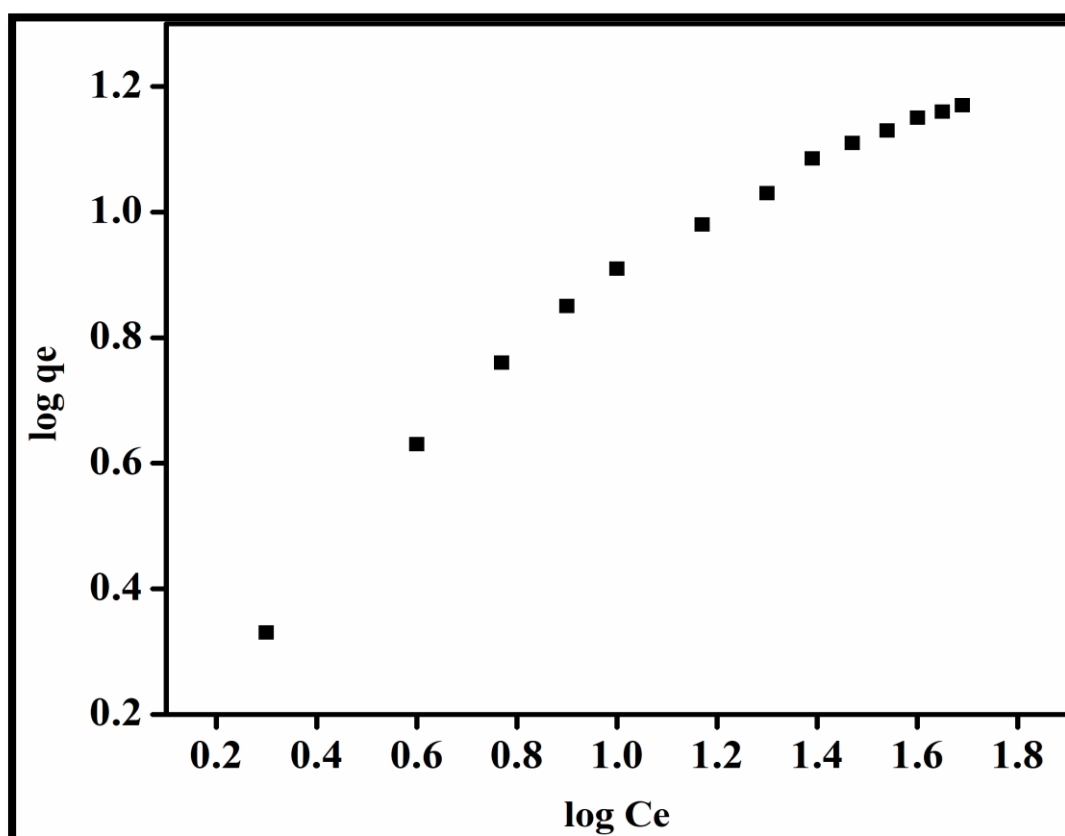


Fig.5.11 Freundlich isotherm graph on CaO-Fe₂O₃ with Zn²⁺

5.3.3 Temkin Model

According to the figure 5.12, multi-layer adsorption will take place when the adsorbent surface increases, the adsorption heat of all molecules decreases linearly, and adsorption is typified by uniform or maximum distribution binding energies. In a similar manner, it was found that the determination coefficient (R²) of the Zn²⁺ ion was 0.994. The values are given below in the table 5.4

Table 5.4 Temkin adsorption isotherm parameters

Adsorbent	K_T (L mol ⁻¹)	$b \times 10^{-3}$ (J g mol ⁻²)	R^2
CaO-Fe ₂ O ₃	0.826	1.601	0.994

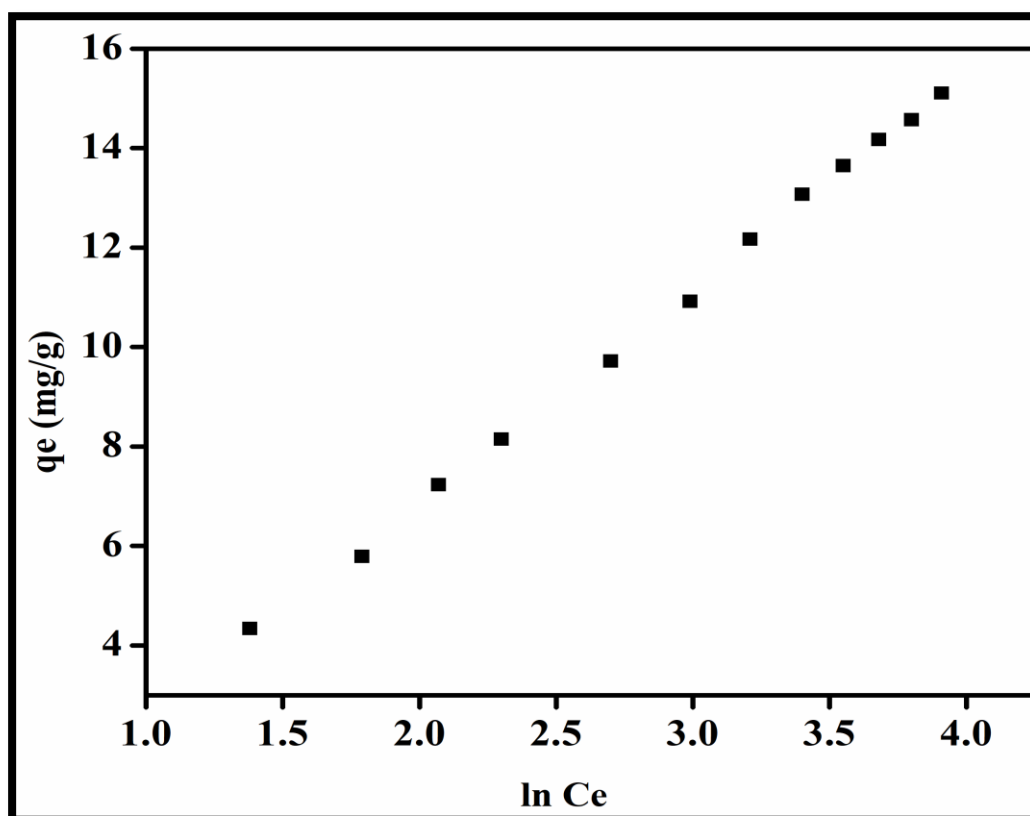


Fig.5.12 Temkin isotherm graph on CaO-Fe₂O₃ with Zn²⁺

5.4 ADSORPTION KINETICS MODEL

5.4.1 Pseudo first order kinetic model

The adsorption capacity as well as the estimated values of $K = 0.039$ and $q_e = 4.998$ are displayed in the image (Fig.5.13) and Table 5.5. The values of R^2 in this pseudo first order equation imply the least amount of elimination, and as a consequence, the pseudo first order cannot be satisfied by the data. As a

consequence of this, this mechanism takes place during the process of physisorption.

Table 5.5 Pseudo first order kinetic model parameters

Adsorbent	Kinetic model	K (min ⁻¹)	R ²	qe (mg g ⁻¹) (Experimental)	qe (mg g ⁻¹) (Theoretical)
CaO-Fe ₂ O ₃	Pseudo First order	0.039	0.975	6.68	4.988

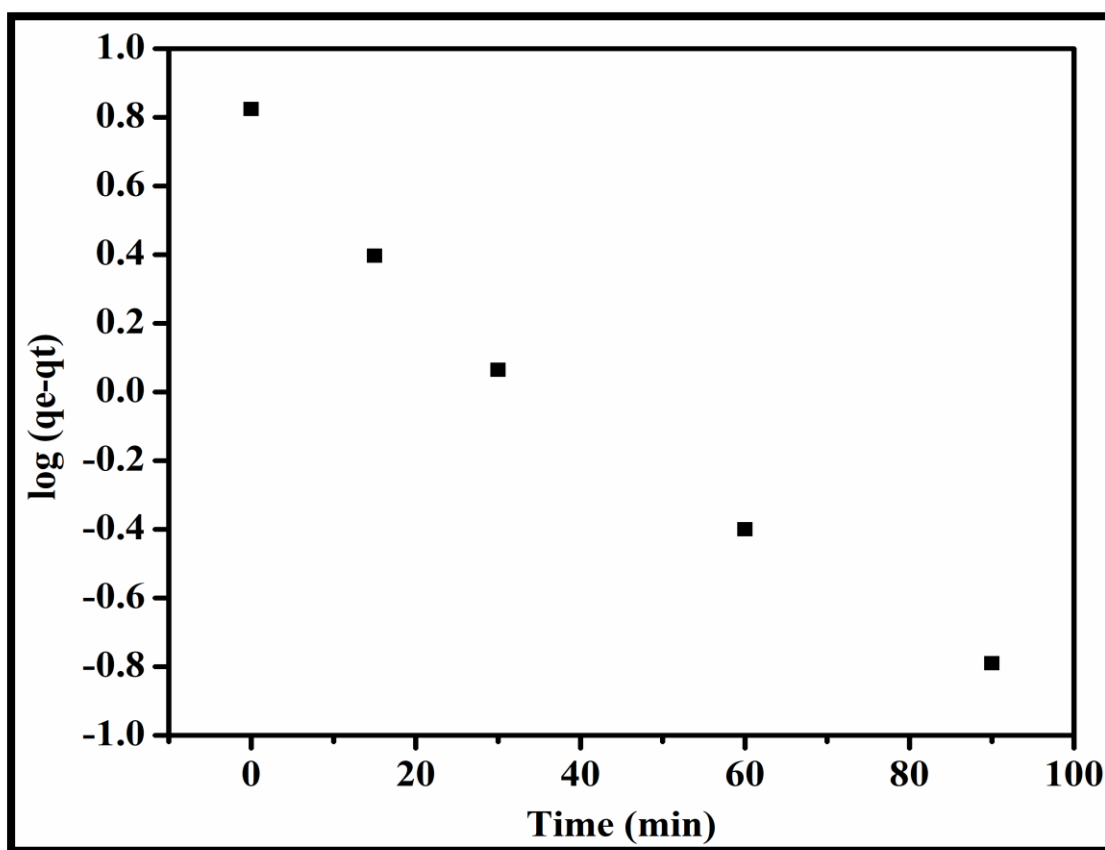


Fig.5.13 Pseudo first order kinetic model graph on CaO-Fe₂O₃ with Zn²⁺

5.4.2 Pseudo second order kinetic model

The adsorption capacity of CaO-Fe₂O₃ is presented by a graph (Fig.5.14) with the values K = 0.025 and q_e = 6.993 displayed in Table 5.6 and calculated respectively. As a consequence of this, the values of R² in this equation of pseudo-

second order indicate a greater amount of elimination, and their values correspond to the pseudo-second order. As a consequence of this, this mechanism takes place during the process of chemisorption.

Table 5.6 Pseudo second order kinetic model parameters

Adsorbent	Kinetic model	K (min ⁻¹)	R ²	qe (mg g ⁻¹) (Experimental)	qe (mg g ⁻¹) (Theoretical)
CaO-Fe ₂ O ₃	Pseudo second order	0.025	0.998	6.68	6.99

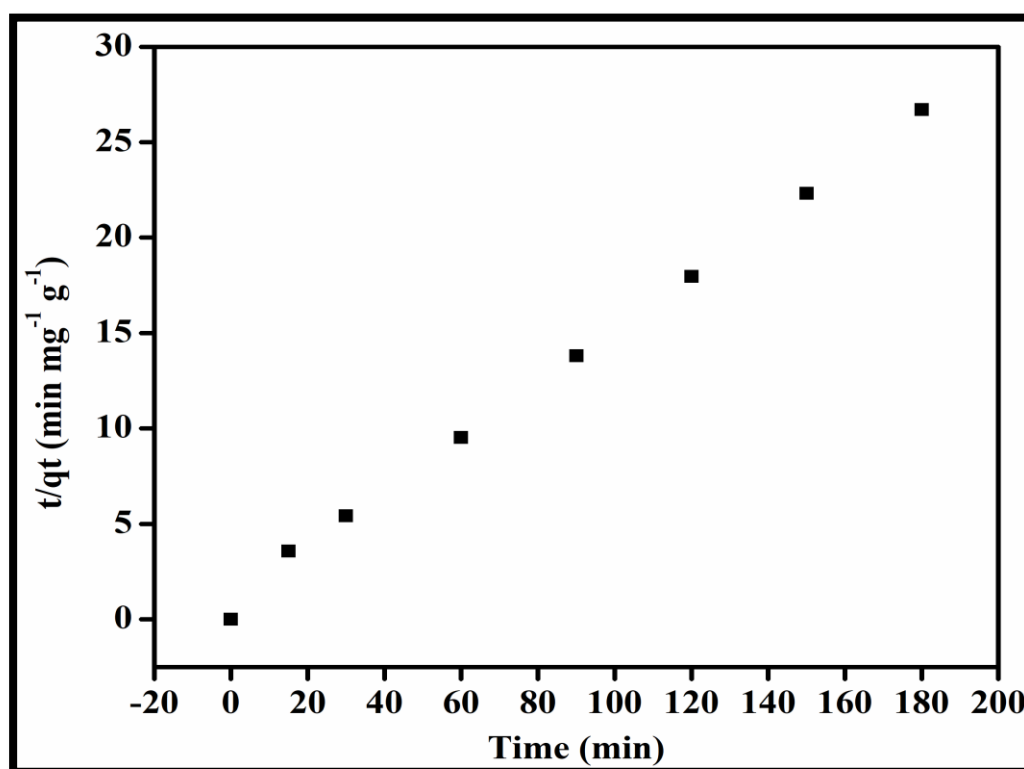


Fig.5.14 Pseudo second order kinetic model graph on CaO-Fe₂O₃ with Zn²⁺

CHAPTER-6 SUMMARY AND CONCLUSIONS

In this study, the removal of Zn^{2+} ions from aqueous solutions was carried out in a batch approach using $\text{CaO-Fe}_2\text{O}_3$ nanocomposite as a novel adsorbent. The method used was batch chromatography. The chemical co precipitation method was utilised in order to fabricate the nanocomposite in question. After that, the impact of a number of parameters, including pH level, beginning concentration of Zn^{2+} ion, contact time, and adsorbent dosage, was investigated. The pH range of 5 was shown to produce the greatest amount of adsorption yield. As the pH rose over this range, hydroxide ions began to precipitate out of solution, which resulted in a reduction in the efficiency of the treatment. In addition, the highest possible adsorption yield was found to be achieved by using a composite concentration of 2 g/L. Under these conditions, it was concluded that a removal efficacy of 97.53% was optimal. In addition, increasing the temperature over 30°C resulted in a reduction in adsorption effectiveness. This indicated that exothermic adsorption of Zn^{2+} ion on the adsorbent occurred, as well as damaged active sites on the surface of the $\text{CaO-Fe}_2\text{O}_3$ nanocomposite. In order to analyse the surface of the adsorbent, FTIR, XRD, EDAX, and FESEM were all utilised as investigative tools. The Langmuir isotherm model was discovered to be superior to the others in the isotherm study due to the fact that it had a greater correlation coefficient (R^2). The maximum amount of adsorption that can occur per unit of weight, as calculated by the Langmuir model, is 60.60 mg/g. A quasi-second-order model was discovered through the study of the kinetics of Zn^{2+} ion adsorption onto $\text{CaO/Fe}_2\text{O}_3$ nanocomposite. The sorption thermodynamic was able to establish the spontaneous and exothermic sorption of Zn^{2+} ion by the $\text{CaO-Fe}_2\text{O}_3$ nanocomposite. In conclusion, several functionalizations could be examined in order to increase the efficiency of the removal process. It has been demonstrated that research into the synthesis, processing, characterisation, and application of nanomaterials for the removal of heavy metals from industrial applications like wastewater treatment or biological systems is likely to bear fruit.

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ABSTRACT

Water contamination is becoming a major problem for us. It is brought on by a variety of hazardous toxins, including heavy metals, dye, and oil spills. It was determined that treating this was necessary in order to reduce water contamination. The removal of Zn^{2+} using the produced adsorbent is investigated. Zinc nitrate is a very poisonous compound that can lead to cancer of the skin, bladder, and lungs as well as destroy aquatic life. Adsorption is favored over all other removal methods due to its lower chemical consumption and large range of adsorbent options. Because of its extremely strong magnetism and recyclability, $CaO-Fe_2O_3$ has been proven to be useful. The large-scale wastewater treatment facilities, home purifiers, and industrial effluent treatment facilities all employ novel adsorbents with excellent adsorbent capabilities to remove Zn^{2+} . Heavy metals, dyes, and many other contaminants are effectively removed using calcium oxide, which has minimal commercial value. The characteristic of magnetic particles is revealed by a number of characterization techniques, such as SEM with EDX, XRD, FTIR, and VSM. In the paper below, "Metal oxide helped by activated carbon nanocomposite utilised to eliminate heavy metal ions, an ecological pollutant created by solution,". The metal oxide - AC nanocomposites were distinguished by XRD, SEM-EDX, FTIR, VSM. An investigation was conducted to determine how successful the nanocomposites were as adsorbents in the process of removing heavy metals from water that comes from nature. The efficacy of heavy metal purification relies on the pH of the solution, is related to carbon content, the dosage of carbon nanocomposites, and the temperature of the nanocomposites, and decreases with initial concentration of the heavy metals. A cheap, recyclable, scalable, and environmentally acceptable adsorbent particle for extracting heavy metal ions from polluted water was identified as the Metal Oxide -AC. For the removal of heavy metals from wastewater, adsorption methods are widely utilised. Adsorption is a cost-effective method that has gained popularity for its little waste disposal benefit. Before to choosing an adsorbent, consider its affordability, technical suitability, and ease of access to raw materials with minimal detrimental effects on the system. The chapter's originality comes from its coverage of a broad range of adsorbents and how well they remove wastewater that contains heavy metals.

CHAPTER-1 INTRODUCTION

The recent enormous gains in industrialization and urbanisation have brought to the forefront, on a global scale, the issue of water pollution as a result of these trends. Significant industrial pollutants consist of dyes and hazardous metals, which are utilised in the production of a wide range of goods, such as textiles, batteries, leather, fertilizer, chemicals, mining, metallurgy, and refining. Other products that contain dyes include leather and textiles. These businesses dump their waste into an environment that already has high concentrations of heavy metal ions and dye, both of which are known to be hazardous to human health. Some of the ailments it can cause include skin irritations and allergies, tumors, headaches, and respiratory problems. It can also cause allergies. Additionally, it is detrimental to aquatic habitats because it reduces the amount of light that can pass through the water. Heavy metals that cannot be broken down by biological processes, such as zinc, cadmium, and copper, nevertheless provide a significant risk to the environment. People should not drink the water since it contains low amounts of heavy metals and other contaminants. They rapidly accumulate inside living organisms, which can lead to infections and problems with metabolism. In addition, the presence of heavy metals poses a significant threat to marine life. Both heavy metals and dye are known to cause substantial damage to ecosystems; hence, it is essential to remove these toxins from sewage in order to safeguard human health as well as the health of the environment. For the purpose of removing dangerous pollutants and heavy metals, numerous treatment processes, such as adsorption, precipitation, membrane filtration, chemical oxidation, ion exchange, and electrolysis, are utilised. Methods that fall within this category include those that are physical, chemical, and biological. Chemical modification of AC, which includes impregnation, base, and acid, has demonstrated to be more effective in eliminating heavy metals and dyes from aqueous solutions as compared to earlier treatment procedures. The ability to absorb of the substance absorbing dye or metal ions increased as a result of the material being processed comprising acid or alkali, along with the presence of activated carbon containing a variety of different metal oxides. Modification of surface functional groups required as well for the adsorption of specific metal ions. This was a prerequisite for the process. Extensive research has been conducted on the subject of the deposition of heavy metals and colours onto chemically modified bio-char. It has not been determined whether chemically modified activated carbons are successful at removing heavy metals and effluent dyes by the use of any review studies that have been conducted. This study focuses on determining the most effective treatment methods for increasing adsorption. We also address the amount of dye and heavy metal that is absorbed by activated carbon as a result of the chemical alterations that are made to it. Zinc, cadmium, chromium, and copper are just a few examples of the heavy metals that can be found in waste products from certain industries. When ingested in large quantities, it can cause headaches, nausea, vomiting, liver cirrhosis, renal troubles, difficulty breathing, and stomach pain. It is also a carcinogen. Before

being released back into the environment, sewage that contains heavy metals that are toxic to living things must first be treated and purified.

In this setting, the study of nanotechnology has been going on for quite some time.

Researchers have also begun using metal oxide nanoparticles as a new generation of adsorbent for the purpose of treating water in order to combat the declining quality of the water supply. Metal oxide possesses a number of useful qualities, including a high surface area to volume ratio, an adjustable surface area, outstanding magnetic properties, biocompatibility, the ability to be easily separated by an external magnetic field, reusability, and a relatively affordable price point. As a result, it makes a fantastic adsorbent. In addition, it has been demonstrated that combinations of activated carbon and iron oxide are effective at eliminating a variety of contaminants from aqueous media. According to the research that has been done, the importance of agricultural wastes extends to the fields of science, society, and the economy. This is due to the fact that agricultural wastes are readily available, do not require regeneration, and only require a straightforward treatment before being thrown away.

1.1 HEAVY METALS AND ITS TYPES

1.1.1 ARSENIC (As)

The presence of arsenic can be found in around 95% of all detergents, dyes, paint, metals, semi-conductors, colors, and pharmaceuticals. In addition, the release of increased quantities of arsenic into the environment may be caused by the use of specific herbicides, fertilizers, and methods for feeding animals. Arsenic (As) is a naturally occurring metalloid that is a component of much more than 245 different minerals. It is the 12th most prevalent component in the human body, the 20th most common component in the crust of the Earth, and the 14th most prevalent component in saltwater. Low-level arsenic exposure can cause damage to blood vessels, which can lead to symptoms such as nausea, vomiting, a prickling sensation in the hands and legs, a reduction in the production of erythrocytes and leukocytes, and irregular heartbeats. People can be exposed to arsenic on a chronic basis through a variety of different pathways, including accidental sources, industrial sources, and naturally occurring resources. Because of arsenic pollution, people in every region are now worried about the state of the environment.

The health of millions of people is put in jeopardy because of the high amounts of arsenic found in river basins, a topic that has received a lot of attention recently. Arsenic is a semi-metal. In its metallic form, it is brittle, bright, and has a tint that is between silver and grey. Arsenic is a notoriously lethal toxin. Arsenic compounds are frequently employed as rat poisons and insecticides, despite the fact that their use is strictly regulated. When exposed to damp air, the fundamental arsenic will tarnish on its surface. When it is burned in the presence of oxygen, it produces an aroma similar to that of arsenic trioxide and burns with a flame that is bluish-white and very bright. Cancer of the skin is one of the side effects of exposure to heavy metals containing arsenic. Lung and bladder cancer are also possible outcomes of prolonged exposure. In addition to recommending that people avoid drinking water that contains arsenic because it is hazardous to their health, the International Agency for Research on Cancer (IARC) established arsenic and its derivatives as substances that might cause tumours. It is strongly recommended that arsenic concentrations in drinking water not exceed 0.05 mg/L at any point.

1.1.2 LEAD (Pb)

Lead is a toxic environmental contaminant. It causes severe damage to a variety of organs in the human body. Lead can sometimes be absorbed through the skin, but the digestive and circulatory systems are often the primary pathways through which it is taken in. Exposure to lead can result in major difficulties with the bronchial system, the genital tract, and the heart due to the metal's high resistivity, peroxidation, and systemic inflammation.

The legal limitations for the quality of water cannot be exceeded when lead ions are present in wastewaters from industrial processes at quantities ranging from 200 to 500 mg/L. Low concentration lead treatments in plants induce severe physiological processes in their capacity for photosynthetic energy, which in turn leads to strong inhibition of plant development. This is because of the tremendous instability in ion absorption that low concentration lead treatments cause. The main component in the ionic process of lead poisoning, which will eventually have an effect on the cell metabolism of live

cells, is the ability of lead metal ions to substitute with other bivalent (Ca^{2+} , Mg^{2+} , and Fe^{2+}) as well as monovalent (Na^{+}) cations. This capacity is what gives lead metal ions the ability to cause lead poisoning. In the end, this effect is what leads to lead poisoning.

Ionic lead poisoning has an effect on all of these processes, as well as cell adhesion, intracellular and intercellular communication, ionic transport, enzyme control, and neurotransmitter release.

These are the qualities of their physical appearance.

A few of its properties included a low melting point, low strength, an easy manufacturing process, chemical resistance, electrical as well as chemical reactivity with sulfuric acid, chemical resistance in air, water, and soils, and the ability to absorb sound waves, nuclear radiation, and shock waves. The metal lead has a brilliant appearance and a bluish-white tint. It is quite pliable and elastic to the touch. The amounts of lead that are typically found in drinking water are 0.01 mg/L.

1.1.2 MERCURY (Hg)

Mercury on Earth has deteriorated due to the increased sensitivity, quality, and diversity of acute heavy metal poisoning. This change has been for the worse. Mercury, which is a silvery-white metal, will ultimately degrade and solidify into a pliable lump if it is exposed to humid air. It is a neurotoxic molecule that results in harmful effects to microtubules and mitochondria, as well as lipid peroxidation and the accumulation of other neurotoxic chemicals.

Mercury is released into the environment as a byproduct of activities in a variety of industries, including the pharmaceutical business, the paper and pulp preservatives sector, the agriculture industry, and the industry that produces chlorine and caustic soda. It is recognised as one of the heavy metals that contribute to the degradation of the natural environment. If the kidneys, brain, or developing foetus are exposed to metallic, organic, or inorganic mercury, all three types of mercury can cause damage to those organs.

Mercury and other elements, when combined, can combine to generate both organic and inorganic forms of the element mercury. Agriculture, municipal

wastewater discharges, industrial sewage discharges, and incineration are the primary human activities that contribute to mercury contamination. Other human activities that contribute to mercury contamination include mining. It is a naturally occurring metal that is odorless, brilliantly white in color, and has no metallic lustre. When heated, it will transform into a gas that has neither a smell nor a colour. Calculations indicate that a total of no more than 2,200 metric tonnes of mercury might be released into the environment every single year. Organic mercury poisoning can be difficult to detect since its symptoms, such as stress, headaches, tremors, weariness, hair loss, and memory issues, are similar to those of other illnesses. These symptoms include stress, headaches, tremors, exhaustion, and hair loss. It has a significant potential for bioaccumulation and is hazardous in its natural state. There can be no more than 0.001 milligrammes per litre (mg/L) of mercury in water that is suitable for human consumption.

1.1.4 CADMIUM (Cd)

Cadmium has emerged as a key metal that is employed in the production of nickel-cadmium (Ni-Cd) rechargeable batteries as well as an anticorrosion coating for steel and iron. Cadmium has also found use in the medical field.

During World War I, cadmium was utilised for the very first time in the place of tin in the manufacturing of paint as well as in the capacity of a tint. In modern times, it can be discovered in the production of several goods, including tobacco smoke, rechargeable batteries, some types of alloys, and more.

Cadmium is utilised in numerous products manufactured in the current day, including batteries, coatings, alloys, solar cells, pigments, plastic stabilizers, and coverings (electroplating). It is also utilised in the production of plastic stabilisers. It has been demonstrated that cadmium can be found in seafood, wheat, and rice. Cadmium can also be found in some drinking water.

The lungs are cleaned of cadmium by breathing in dust from commercial settings. In industrial settings, being exposed to cadmium, whether for a short period of time or for a longer period of time, can be harmful to the kidneys and induce lung illness. The levels of cadmium in the blood of smokers are

approximately twice as high as those of non-smokers. Cadmium levels in drinking water are allowed to reach a maximum of 0.03 mg/L.

1.1.5 CHROMIUM (Cr)

The crust of the earth and the ocean are both sources of chromium (Cr), a naturally occurring heavy metal that is utilised in various manufacturing processes. The most common and stable forms of chromium have been determined to be trivalent and hexavalent, with oxidation states ranging from +2 to +6. The name "chromium," which derives from the Greek word *chromes*, which means "color," alludes to the toxic hues that can appear in a range of intensities. The word "chromium" originates from the Greek word *chromes*.

Chromium (Cr) is an example of a complex ion that is part of Group VI of the Periodic Table. It increases an alloy's resistance to corrosion and lengthens its durability.

Chromium is a metal that is difficult, brittle, and shiny. It has a tint similar to silver grey and is very easy to polish. Because it produces and develops green chromic oxide when it is heated, it does not oxidise when it is exposed to air. Due to the metal's sensitivity to oxygen, chromium soon produces a thin oxide coating that acts as a shield and protects it from the element. Heavy metals can accumulate in the body when chromium is present, which can lead to a variety of health problems. Hypothyroidism is a condition that can impact a person's skin, kidneys, neurological system, and digestive system, among other body systems. Other disorders that impact these systems include the development of many cancers in the lungs, throat, stomach, liver, testicles, and spine. Other diseases that affect these systems include diabetes and heart disease. The maximum chromium concentration that can be present in drinking water is 0.1 mg/L.

1.1.6 COPPER (Cu)

Copper (Cu) is an element that occurs naturally and is characterised by a reddish-brown colour and a high degree of malleability in its purest form.

The most common applications for copper can be found in industrial machinery, roofing, plumbing, and electrical wiring. The vast majority of these applications require the usage of copper in its purest form.

However, in order to attain higher degrees of hardness when it is required to do so, it can be alloyed with a number of other metals. Heavy metals are known to have deleterious effects on a range of human organs, both in the immediate future and further out into the distant future. The toxic effects of heavy metals can cause a variety of adverse health impacts, including cancer, genetic diseases, issues with the immune system, issues with the digestive and urinary systems, issues affecting the central nervous system, lesions, ischaemia. Copper has the ability to both speed up and slow down the oxidation process.

Copper plays a significant role in the human metabolism because it assists in the normal functioning of a wide variety of fundamental and vital enzymes and contributes to the creation of haemoglobin, myelin, and melanin. Copper also helps regulate blood sugar levels. It is necessary for the biomolecules' structural as well as metabolic cofactors in order for biomolecules to function properly. As a transition metal that signals both within and outside of living cells, as well as among live cells themselves, copper plays an essential part in the transmission of information both inside and outside of the brain. The recovery of heavy metals in general, and copper in particular, is a topic that is receiving a lot of attention from the general public because of the positive effects that removing pollutants from industrial effluents may have on the environment and the economy. The maximum allowable amount of copper that can be found in drinking water is now 1.3 mg/L.

1.1.7 NICKEL (Ni)

Nickel, a brittle and silvery-white metal, can solidify into formations resembling cubes. It is also resistant to corrosion and features ductility and malleability in addition to high strength. In point of fact, researchers have discovered that each cigarette contains anywhere from 1.1 to 3.1 grammes of nickel. It is possible that this metal is present in the smoke in the form of nickel carbonyl, which is exceedingly detrimental to one's health.

Nickel can also enter individuals' bodies through the consumption of certain foods. In point of fact, a wide variety of foods, such as cocoa, chocolate, and almonds, as well as spinach, asparagus, carrots, broccoli, and green beans, contain considerable amounts of this potentially harmful element.

Nickel is mistakenly consumed when food is cooked on stainless steel appliances or when food comes into contact with the mouth. In addition, nickel can be absorbed via the skin. Nickel and nickel compounds are common examples of hazardous substances that can be found in industry. It is well known that nickel and nickel compounds can have an effect on people even when they are not exposed to them in the workplace. Because it is found in air, food, and water, nickel is accessible to the whole population and can be absorbed by them. Regarding the potential adverse health effects of occupational nickel exposure, inhalation is a significant route that must be considered. Even though the majority of nickel found in the human body comes through food and drink, the inability of the intestine to absorb nickel makes this route far less important. Those who are subjected to nickel may experience a variety of adverse health impacts, including allergies, conditions affecting the kidneys, heart, and lungs, fibrosis, and cancers of the lung and nose. The maximum concentration of nickel that can be found in drinking water is 0.1 milligrammes per litre.

1.1.8 ZINC (Zn)

Zinc is a metal that has a dazzling bluish-white appearance.

The element can be found in the periodic table's Group II (b) section.

When it is at normal temperature, the material is crystalline and brittle, but when it is heated, it changes into a substance that is ductile and malleable.

Zinc (Zn), one of the most frequent heavy metals in the Earth's crust, is regarded to be an essential compound metal necessary for the growth of living things. This is due to the fact that zinc is a component of a large number of major proteins and governs the regulation of a number of immune-modulatory processes. Zinc is also one of the most common heavy metals in the Earth's crust. However, its toxicity, which will be maintained by a number of human sources, ongoing exposure to contaminated places, and other natural

occurrences, parallels its necessity. The equilibrium between dietary zinc absorption and zinc loss from the body is referred to as zinc's homeostasis, and Zinc is a heavy metal that plays an important role in human health.

Zinc is essential to the functioning of many different biological processes, including those that ensure survival, promote growth, and heal damaged tissue. Due to the high bioavailability of zinc found in a variety of fruits, vegetables, and dairy products, Zinc is a form of heavy metal which is essential to human health and plays a vital function overall.

The most common applications for zinc metal are in the production of brasses and alloys for die casting, as well as the prevention of corrosion in iron and steel. Zinc is more resistant to common atmospheres than iron is, and as a result, it corrodes at a significantly slower rate. Symptoms of heavy metal poisoning in zinc include nausea, vomiting, and diarrhoea. Iron, on the other hand, is more susceptible to corrosion in typical environments. An overdose of zinc can cause a variety of adverse health effects, including metallic oxide fever, digestive and liver problems, and more. The maximum amount of zinc that can be present in drinkable waters is 5.0 milligrammes per litre. This is the safest limit.

There are several different types of magnetic nanoparticles.

1.4.1 Carbon-based nanoparticles as their primary component

The ability of various carbonaceous compounds, such as carbon nanotubes, carbon black, graphene oxide, and biochar, to assimilate various environmental toxins has been the subject of extensive research in recent years. In the treatment of wastewater and water, ²⁴the most commonly used kind of carbon adsorbent is activated carbon. One of the ⁵numerous environmental applications that may benefit from the synthesis of ⁵activated carbon in the type of sorbents in a wide range of forms is the elimination of heavy metals from water-based solutions. In the research of heavy metal adsorption, a novel idea asks for the utilization of biochar instead of activated carbon generated from coal.

This change was brought about by the significant increase in manufacturing costs that are associated with the traditional approach.

Since the development of nanotechnology, a great number of research have been carried out on the potential applications of graphene-based compounds, such as Carbon Nanotubes, reduced Graphene Oxide, and Graphene Oxides, for the removal of hazardous metals.

1.4.2 Nano zero valent ion (nZVI)

Nanoscale zero-valent nanoparticles have emerged as an innovative and extremely effective technology for the remediation of heavy metals. This is due to the vast specific surface area that these nanoparticles possess as well as their outstanding capacity for reduction.

Because of its great reactivity, extraordinary reactive longevity, and low toxicity, nanoscale zero-valent ion (nZVI) has shown exceptional promise for the reduction of heavy metal ions in aqueous solutions. nZVI has been utilised successfully to treat a range of metallic ions in aqueous solutions.

1.4.3 Silica based nanoparticles

Nanomaterials have been the subject of investigation and study by scientists throughout the past decade. SiO₂ has long been regarded as the best material for water treatment due to its unique qualities, which also include financial viability, non-toxicity, great acid tolerance, and ease of modification. Several nanostructured silica materials have been utilised to remove heavy metals. SiO₂ has long been regarded as the finest material for water treatment.

The improved performance in surface group absorption characteristics of SiO₂ was the focus of the initial investigations, and the effect of group type on the sorption of active SiO₂ has been studied only on occasion. It should be noted that the precise amounts of complex chemicals bound to these, as was

stated earlier, modified SiO₂ nanoparticles are unknown. This is because the precise amounts of complex chemicals bound to these nanoparticles have not been studied.

As a consequence of this, it may be difficult, if not impossible, to identify with absolute certainty whether or not particular chemical groups have a higher affinity for the target ion in question.

1.4.4 Nanocomposites

removing impurities from water by utilising nanostructured materials. They feature a high ratio of surface area to volume, which contributes to their excellent attributes, as well as their compact nature, which gives them a huge surface area. These qualities contribute to an increase in the efficiency with which nanostructured materials can be removed. These materials are distinguished from other types of materials because to the extraordinary features they possess, which include a huge specific surface area, a significant level of reactivity, and the capacity to catalyse reactions. Nanostructured particles, because of their high surface area, have a great number of active sites that are capable of reacting with a wide variety of chemical species. There has been a recent uptick in interest in nanocomposite materials, which are produced by combining nanostructured materials with the interlayer space of clay particles. This is because nanocomposite materials possess distinctive physicochemical features and have the potential to find uses in a wide range of industries. The excellent surface qualities of nano-silica were put to use in the production of two innovative composites via a chemical procedure that was straightforward and safe for the surrounding environment. The nanocomposite was evaluated using a variety of different approaches in order to provide evidence for the shift. The nanocomposite was utilised to either absorb or eliminate metals after first being positioned into a glass column.

1.4.5 Nanoparticles Comprised of Polymeric Substances

Over the course of the last 10 years, metallic nanoparticles adsorption (MNP) ⁵³ has received a great deal of attention as a result of its efficiency as well as the extensive number of uses it might have. Because carrying out the treatment won't require much effort on your part. Surface polymeric MNPs, which includes such MNP polymer composites with core-shell nanoparticles, ⁵⁷ have ¹⁶ recently attracted a great deal of interest. This is due to the fact that the polymer shell prevents the core portion of the Magnetite nanoparticles from collapsing into bulk materials and improves the dispersion stability of the core-shell nanoparticles in a suspension medium. The organic and aqueous phases are both capable of successfully distributing these polymeric nanoparticles. It is possible to speedily synthesise polymeric nanoparticles by employing a straightforward precipitation method and low-cost starting ingredients.

1.4.6 Ceramic Nanoparticles

Ceramic membranes are becoming increasingly used in a wide number of applications due to the large chemical and temperature operating range that they offer. The robustness of the ceramic membrane is an important consideration throughout the application process. It has been exploited in the process of oil extraction from waterways as well as the cleaning of effluent from the dyeing process. Ceramic membranes' remarkable antifouling properties and long operational life greatly minimise the high startup design complexity. A new area of research that uses a ceramic barrier for the removal of pollutants has very few published studies. Ceramic water filters are utilised in over 20 countries as an alternative point-of-use water purification system. Although ceramic membranes have a number of benefits, their usage in large-scale applications for metallic ion-contaminated effluent is limited because of the greater cost of membrane fabrication. Ceramic membranes have been shown to be effective at removing metallic ions from water. Clay and trash are the two materials that are gathered from the surrounding area

and used in the manufacturing of ceramic water filters. In comparison to polymeric composite membranes, ceramic composite membranes have superior antifouling properties and greater mechanical and chemical durability. It has been demonstrated that CWFs can effectively eradicate viruses larger than 1 metre in diameter by removing nearly 99 percent of particles larger than 1 metre in diameter. Ceramic composite membranes also have superior antifouling properties than polymeric composite membranes.

1.4.7 Metal Oxide Nanoparticles

According to research, surface effects have a significant impact on the magnetic properties of iron oxide nanoparticles. Fe₃O₄ nanoparticles (MNPs) are a new class of nanomaterials that have the potential to be used in environmental cleaning. Iron-based magnetic nanoparticles have distinctive characteristics, such as a greater surface-to-volume ratio, a reduced demand for chemicals, and the absence of secondary pollutants. Direct silylation of magnetic particles in order to produce Fe₃O₄ adsorbents is accomplished through the use of cross-linking agents, organic gas precipitation, polymer coating, and surfactant adsorption the vast majority of the time. The scientific community has shown a significant amount of interest in materials that have nanoscale-specific features. Magnetic Fe₃O₄ nanoparticles are an intriguing new category of nanometer-sized components because they can exhibit a variety of distinct and specified properties. These properties include a large surface area, the possibility for self-assembly, high precision, chemical sensitivity, catalyst potential, and the absence of internal diffusion resistance. These properties hold great promise for the development of new or improved wastewater treatment innovations. Because of their superparamagnetic nature, these particles are drawn to a magnetic field; however, once the field is removed, they lose all of their magnetic qualities. Magnets can be used to remove pollutants, and the super-paramagnetism supplied by Fe₃O₄ nanoparticles is an added benefit.

4 Heavy metal removal technology

The dangers of having heavy metals in the food chain as well as within our bodies have been discussed in greater depth in the portions of the thesis that came before this one. Because of this, it is of the utmost importance to treat wastewater and remove as many heavy metals as is reasonably practicable from it before releasing the wastewater back into the environment. Heavy metals have risen to the top of the list of most concerning environmental pollutants over the course of the last few decades as a result of the development of more stringent rules and regulations governing the treatment and disposal of wastewater. As a result, the technologies that can remove heavy metals from wastewater are of considerable interest and significance. There are a variety of methods and technologies available for removing heavy metals; however, the three applications that are used the most frequently are described below.

The release of chemicals

Chemical precipitation is the removal method that is used most frequently in wastewater treatment plants (Fenglian Fu, 2010). This is primarily due to the ease with which it can be carried out. The solubility (K_{sp}) of the metals involved, the pH of the wastewater, the concentration of the metals in the produced wastewater, and the presence of a variety of other substances can all have an effect on the precipitation factor of the metal. Precipitation can also be affected by the concentration of the metals in the produced wastewater. Depending on the chemicals that were used for the precipitation process, a utilised chemical reacts with the ions of heavy metals that are present in the effluent to generate insoluble precipitates of the heavy metals. These precipitates can take the form of hydroxide, carbonate, sulphide, or phosphate. The generated precipitates are subsequently removed as residue after going through the processes of coagulation, flocculation, sedimentation, and filtration. Alterations in parameters like pH, temperature, conductivity, and metal concentrations can be utilised to improve the rate at which metal ions

are removed from a solution. The substantial quantity of concentrated and poisonous sludge that is created necessitates extra treatment as well as disposal. Despite the fact that the procedure is very basic, effective, and can be cost-effective when compared to alternative removal technologies, the disposal and treatment of the produced sludge is necessary.

Ion Exchange

The process of eliminating heavy metals through ion exchange is one of the more easy options available. The process, as its name suggests, involves exchanging the metal ions that are present in waste water with the ions that are present in an ion exchanger that is either synthetic or natural. Zeolites are the natural ion exchangers that are used more frequently than any other type. Synthetic ion exchangers are favoured over natural ones (Fenglian Fu, 2010) due to the former's superior efficiency and the latter's ability to extract the vast majority of the targeted metal ions from the latter's solution. The positively charged, potentially toxic metal cations that are affecting the purity of the water, such as zinc, nickel, and copper ions, are exchanged with the positively charged ions from the resins, such as hydrogen and sodium ions, in equivalent amounts in the solution. The same is true for the negatively charged unwanted anions that are present in the wastewater solution, such as hydroxyl anions. Ion exchangers are insoluble solid substances. The polymers can be categorised as either cation or anion exchangers, and this designation is determined by the charges of the ions that are being traded. (IWA publishing, 2011). The approach is particularly sensitive to a variety of parameters, such as pH and temperature, as well as the concentration and duration of contact with the metal. (Fenglian Fu, 2010). Ion exchange procedures are only suitable for the treatment of heavy metals at low concentrations. Despite their many benefits, including excellent treatment and removal efficiency as well as cost effectiveness (low cost and operating materials), ion exchange methods cannot treat higher concentrations of heavy metals. (IWA publishing, 2011).¹⁵

Adsorption

The remediation of wastewater that is contaminated with heavy metals also makes use of a removal process called adsorption. Adsorption is the process that takes place when the solute in the liquid phase of polluted effluent accumulates on solid or liquid absorbents to form a molecular film. This can take place on either solid or liquid absorbents. (Lakherwal, 2014). In order to successfully remove metals from effluent water, numerous absorbents, such as activated carbon, carbon nanotubes, metal oxides, and a wide variety of agricultural and bio absorbents, have been demonstrated to be effective. According to IWA publishing (2011), activated carbon is still the most common type of absorbent due to the fact that its removal rate of some metal ions is greater than 99.9%. Activated carbon is an effective method for eliminating heavy metals; nevertheless, researchers are constantly working to bring down the cost of the process. Because commercial carbon sources are dwindling, the price of carbon absorbents is climbing every day, and activated carbon materials cannot be recycled, it is essential to find substitutes that do not affect the quality of the final product. Because of its low cost and its ability to treat metal-contaminated wastewater in a natural way, bio adsorption is quickly becoming an increasingly popular treatment method. It has been established that bio adsorbents, which can be either living or dead biomass, can remove heavy metals from wastewater and sludge. These adsorbents can be obtained for a low cost and are easily available. In general, there are three types of sources for biomass: 1) non-living biomass, 2) biomass derived from algae, and 3) biomass derived from microbes. Because of its widespread availability, high treatment efficiency, low cost, and high metal sorption capacity, algal biomass has become the most popular option out of the three, and it is also the one that is now garnering the most attention. (Fenglian Fu, 2010).

1.6 APPLICATIONS OF MAGNETIC NANOPARTICLES

- ❖ Industrial applications
- ❖ Medical and Biomedical applications
- ❖ Catalysis
- ❖ Environmental usage
- ❖ Molecular Imaging
- ❖ Magnetic Separation

1.7 CHARACTERIZATION OF MAGNETIC NANOPARTICLES

- ❖ Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy (SEM-EDX)
- ❖ Fourier Transform Infrared Spectroscopy (FTIR)
- ❖ X-Ray Diffraction (XRD)
- ❖ Vibrating Sample Magnetometer (VSM)
- ❖ Transform Electron Spectroscopy (TEM)
- ❖ Thermogravimetric Analysis (TGA)
- ❖ Braunauer-Emmett-Teller (BET)
- ❖ Atomic Absorption Spectroscopy (AAS)

AIM AND SCOPE OF THIS CHAPTER 3

The purpose of this thesis is to create a unique nanocomposite for the removal of zinc from waste water that has improved capacity and is easy to use, cost effective, and friendly to the environment. The goals of this study were to (1) generate metal oxide nanocomposites, (2) apply the obtained nanocomposites to the adsorption of zinc in water, and (3) explore the process of adsorption of these synthetically produced materials.

Development of nanocomposite materials

The manufacture of calcium oxide that had been impregnated with Fe_2O_3 was the first step in this investigation. Next, the material was characterised. Nanoparticles made of iron oxide that have been synthesised and are magnetic. These nanoparticles are designed to bind heavy metal ions and have coordinating or chelating groups on their surfaces. Magnetic measurements will be used to examine the nano composites in order to assess the separation efficacy of the magnet, which is necessary in order to determine whether or not this removal system will be successful. Following

this step, the nanocomposites will be utilised in adsorption tests with the purpose of removing zinc. If the nanocomposites were put directly to waste water, they would be more effective. This is especially true when one considers the fact that waste water is typically held in containers in order to prevent the sewage system from becoming overloaded. During the process of zinc removal, this would prevent the problems that are caused by a high concentration of suspended matter. The inquiry makes use of calcium oxide, which is a type of metal oxide. Calcium oxide, often known as CaO, has a large capacity and performs exceptionally well as an adsorbent for a wide variety of hazardous compounds. As a result, the purpose of this study is to design a method that is efficient, uncomplicated, and sensitive for extracting zinc from aqueous solutions by utilising nanocomposite calcium oxide (CaO), as well as to discover the circumstances under which this method is most effective. An examination of the adsorbent's surface shape, as well as its chemical and physical properties, was carried out with the assistance of XRD, FTIR, and SEM/EDX.

Adsorption experiments and studies

⁵ The removal of heavy metals can be accomplished using a variety of processes, including chemical oxidation and advanced oxidation, chemical precipitation, chemical coagulation, chemical stabilization, and ion exchange. Nanotechnology has been exploited in recent efforts that have been made to purge effluent of heavy metal ions. Adsorption and membrane separation are the two methods that are used the most frequently. For the purpose of effectively removing heavy metals, numerous nanomaterials, including those based on carbon, metal oxides, zerovalent nanoparticles, and nanocomposites, have been produced. The high removal capacity and selectivity of these metal oxide nanoparticles are two of the many reasons why they are advantageous. The effect of a number of parameters, including the pH of the solution, the initial concentration of chromium, and the amount of adsorbent used, was investigated. After each test, the CaO/Fe₂O₃ nanocomposite adsorbent was removed from the aqueous solution, and the concentration of Zn ions in the solution was measured. We performed the calculations necessary to determine the adsorption capacity of Zn ion as well as its removal efficiency from aqueous solution. The efficacy of the process can be mostly attributed to the type of adsorbent that is used, which is dependent on the concept that the process will result in a net buildup of metal at the surface of the solid phase (adsorbent). The process of adsorption can be broken down into three distinct phases: the transport of the adsorbate from the liquid phase to the solid adsorbent surface, the adsorption of the analytes onto the solid adsorbent surface, and the transit of the adsorbate within the adsorbent particle.

⁴ As a result, this article discusses the processes of adsorption as an efficient way for eliminating hazardous metal pollutants from wastewater.

The process of adsorption

It is usual custom to use the adsorption isotherm that forms within the adsorbate & the adsorbent to analyze the adsorption mechanism. An adsorption isotherm is a useful way of representing the phenomenon that controls the retention (or release) or mobility for a chemical from an aqueous medium to a solid-phase while maintaining constant temperature and pH. In overall, an adsorption isotherm provides a good way to describe the process. Adsorption isotherms are created using the data from experiments that was collected. The most frequent models used to define the method of adsorption onto solid surfaces are the Langmuir and Freundlich models. These models can be used to assess many different adsorbate concentrations, allowing them to deliver information that is useful in the design process and essential for scaling. The Langmuir model predicts that heavy metal adsorption occurs in the form of monolayer on a homogeneous surface due to the equilibrium balance of metal ions among the phases of liquid and solid and the non-linear shape. This is due to the non-linear nature of the model.

Before nanomaterials can be used in the process of removing heavy metals from effluent, a great deal of study needs to be carried out first. This is despite the fact that the usage of nanomaterials has a lot of untapped potential. For instance, the vast majority of research utilise samples of simulated wastewater that have been created in a laboratory. These samples are significantly more straightforward than genuine wastewater. The actual wastewater had a significant amount of organic matter, which increased the likelihood of nanoparticles being blocked and decreased the efficiency of the removal of heavy metals. Research on the actual effluent produced by the treatment plant is very much wanted. The purpose of this research endeavour is to examine how an iron oxide-impregnated calcium oxide nanocomposite operates for the efficient removal of heavy metals from real-world environments.

CHAPTER-4 MATERIALS AND METHODOLOGY

4.1 MATERIALS

The List of materials employed to synthesize Cao-Fe₂O₃ are listed below:

4.1.1 CHEMICALS REQUIRED

- ❖ Hydrochloric Acid (HCl)
- ❖ Ferric Chloride (FeCl₃)
- ❖ Ferric Sulphate (Fe₂(SO₄)₃)
- ❖ Calcium Oxide(CaO)
- ❖ Sodium hydroxide (NaOH)
- ❖ Distilled Water
- ❖ Ammonia solution (NH₃)

4.1.2 EQUIPMENTS REQUIRED

- ❖ Muffle Furnace
- ❖ Hot Air Oven
- ❖ Hot Plate Magnetic Stirrer
- ❖ Rotary Shaker

4.2 METHODOLOGY

Over the course of the past few years, nanostructured metal oxide has established itself as the best adsorption candidate for the treatment of wastewater. Nanoparticles made of metal oxide have a number of properties in common with other nanomaterials, including large pore volumes and surface areas, high adsorption capacities, pores of a good size, and sites that can be functionalized. Iron has the position as the fourth most abundant element found in the crust of the Earth. These nanomaterials have been the focus of a significant amount of research as a direct result of the ease and accessibility with which iron oxides may be produced. They outperform other adsorbing substances in terms of their capacity for adsorption, their affinity for the material being adsorbed, and their rate of adsorption. Iron nanoparticles are frequently used in the process of removing hazardous metals from water and wastewater. Chemical precipitation was used to create the nanocomposite of CaO and Fe₂O₃, which was then characterised.

CaO/Fe₂O₃ nanocomposite synthesis

It has been determined how to make a solution with a molecular ratio of 1:2 that includes both iron (III) and iron (II). Ferric chloride and ferric sulphate are two chemicals that we use in the course of our research. Following this step, roughly 1 g of prepared CaO was added to the mixture, and it was agitated for a total of 20 minutes until the iron ions formed bonds with the CaO particles. After bringing the temperature of the solution up to between 80 and 90 degrees Celsius, thirty millilitres of NH₄OH were added to the combination in a drop-by-drop fashion, and then the mixture was permitted to sit at the designated temperature for half an hour. The manufactured magnetic composite has been isolated from the solution by using filter paper to make the separation. After that, the composite that had been created was allowed to dry at a temperature of 150 degrees Celsius for two hours before it was put to use as an adsorbent to get rid of Zn (II) ions.

Putting together a stock solution of zinc (II)

Deionized water that had been distilled twice was used in the preparation of the Zn (II) ion stock solution. By dissolving 29 grammes of Zn(NO₃)₂·6H₂O in double-distilled water, a stock solution with a concentration of 100 mg/L was produced. Other stock solutions with concentrations of 20, 40, 60, 80, and 100

mg/L were created using this stock solution as a starting point and then diluted with double-distilled water.

Adsorption experiments and studies

During the adsorption experiments, flasks containing 250 milliliters and 100 millilitres of a solution containing zinc (II) ions were loaded with CaO/Fe₂O₃ nanocomposite as an adsorbent. The effect of a number of parameters, including solution pH (which was explored at 2, 3, 4, 5, 6, and 7), starting chromium content (which was researched at 20, 40, 60, 80, and 100 mg/L), and adsorbent dosage (which was studied at 0.3, 0.4, 0.5, 0.6, and 0.7 g), was investigated. Following each experiment, the CaO/Fe₂O₃ nanocomposite adsorbent was removed from the aqueous solution, and the amount of Zn (II) ion that remained in the aqueous solution was calculated. The following equation for mass balance was used to determine the adsorption capacity of the Zn (II) ion in aqueous solution as well as the removal efficiency of that ion.

$$\% \text{ Adsorption} = \frac{C_i - C_e}{C_i} \times 100$$

where C_i denotes the initial Zn (II) concentration in mmol L⁻¹ and C_e denotes the final Zn (II) concentration in mmol L⁻¹.

The following equation was used to compute the quantity of Zn (II) adsorbed (mg g⁻¹) at time t :

where C_0 and C_t are the initial and final Zn (II) concentrations (mg L⁻¹), V is the volume, and $m(g)$ is the weight of the adsorbent.

Isotherms of adsorption

Adsorption is often characterised by the adsorption isotherm among adsorbate and adsorbent. An adsorption isotherm, in general, is a useful tool for defining the phenomena that regulates the retention as well as movement of a chemical from aqueous medium to solid-phase under steady temperature and pH. The experimental data is used to create adsorption isotherms. The two basic models used to define the process of adsorption on solid surfaces are Langmuir and Freundlich. Such models can be utilized to a wide range of adsorbate concentration to offer important information for design & scaling. According to the Langmuir model, heavy metal adsorption occurs as a

monolayer on a homogeneous surfaces due to an equilibrium distribution of metal ions throughout both liquid and solid phases, with the non-linear form given by equation.

The highest adsorption capacity corresponding to site saturation is denoted by $q_{\max}$ (mg g⁻¹), the state of equilibrium metal concentration is denoted by C_e (mg L⁻¹), and the Langmuir constant is denoted by k_L (L mg⁻¹). The Langmuir isotherm indicates that metal ions are chemically adsorbed, with each site containing only one adsorbate, all sites being energetically equivalent, and no ion interactions occurring.

By charting C_e/q_e versus C_e , the equation may be transformed to the corresponding linear form, which can then be used to linearize the experimental data:

The Freundlich model, an empirical model, requires the presence of preferred sites within the adsorption and can be stated in non-linear equation form.

where q_f is the quantity of the adsorb adsorbed in mg g⁻¹ & C_f is the adsorbate concentration at equilibrium.

By calculating the logarithm of each side of the equation, the Freundlich equation may be linearized: k_f (mg g⁻¹) is the Freundlich constants related to adsorption capacity, and $1/n$ is the heterogeneity factor:

Kinetic equations for adsorption

Kinetic equations for adsorption

The method of adsorption mechanisms generated from batch reduction of metal ions empirical data are analysed and interpreted using kinetic models. Kinetics is a movement or variation in the the concentration of the product or reactant over time, also referred to as reaction rate. The dynamics of adsorption kinetics explain how quickly the adsorbate is adsorbed onto the adsorbent, influencing the time at the particle-metal interface. To investigate the mechanism of sorption, two kinetic models were used: (i) a pseudo-first-order kinetics model ; (ii) a pseudo-second-order kinetic model. Lagergren's (pseudo-first-order) model is expressed as follows and is reliant on the amount of metal ion adsorption multiplied by one:

When the original criteria for the definite integration are applied, $q = 0$ at $t = 0$ and $q=qt$ at $t=t$.

The equation is linearized as follows:

wherein k_1 is the pseudo-first-order adsorption rate constant (min^{-1}) and q_e and q_t are the Zn (II) adsorption capacities (mg g^{-1}) at equilibrium and time $t(\text{min})$. The pseudo-second-order model, which is based on the percentage of metal ions on the adsorbent's surface at time t and the amount deposited at equilibrium adsorption enhanced to the second power, is expressed as follows:

When the beginning conditions for definite integration $q = 0$ at $t = 0$ and $q=qt$ at $t=t$ are applied, the equation is linearized to

wherein k_2 represents the pseudo-second-order adsorption rate constant ($\text{gmg}^{-1} \text{min}^{-1}$). The mechanism of metal adsorption upon adsorbents is divided into four major steps: (a) Bulk diffusion of Zn^{+} ions through the solution through the boundary layer at the adsorbent's surface. (b) migration of metal ions (Zn^{+}) through the boundary layer towards the surface of the adsorbent via film diffusion (c) transferring metal ions between the surface to the internal pores of the particles (intraparticle diffusion). (d) Adsorption of Zn^{+} ions using active sites of the adsorbent surface created by chemical reactions that include ion exchange, complexation.

RESULTS AND DEBATE

The overriding purpose of this component of the thesis was to find and exploit nanomaterials that had recently been developed or were currently available on the market. When it came to heavy metal removal from aquatic systems, the nanoparticles were compared to some regularly utilised adsorbents. The dissolvability for both readily accessible metal oxide and lab-made nanoparticles was studied. The rate during which nanoparticles of metal oxide dissolve can be useful information for toxicity researchers. The impacts of exploring various properties on the process of adsorption among heavy metals like Zn(II) are examples of systems studied, and the results are shown below. Contact time, starting ion concentrations, dose, and solution pH are all examples of these parameters.

The surface morphology of nanomaterials

There are currently a large range of materials used for heavy metal removal from aqueous systems, as well as rising qualities that may either rehabilitate or eradicate heavy metals. Considering the fact that certain materials have been shown to be beneficial, they have some disadvantages. The surface formulation of nanoparticles is a significant topic when examining the possible effects of widely accessible and lab-made nanoparticles for utilization in

biological applications. When nanoparticles interact with other cell types, they have the potential to cause cytotoxicity in human or animal cells.

FESEM/EDX

The results of an investigation employing FESEM/EDX on the surface morphology and elemental distribution of a CaO/Fe₂O₃ nanocomposite are shown below.

In the process of adsorbing Zn(II) ions, the surface hardness and particle size of the CaO/Fe₂O₃ nanocomposite can vary, which is something that is both highly significant and effective. After Zn(II) adsorption, the surface of the CaO/Fe₂O₃ nanocomposite has altered. This may be attributed to the substitution of the ions on the nanocomposite surface with Zn(II) ions, which caused the surface to undergo the change. Investigations using FESEM and EDX revealed the presence of Zn(II) ions in the structure of the magnetic composite material. The findings of these investigations also showed that the surface of the substance contained carbon, oxygen, iron, and calcium in an even distribution. The formation of magnetic nanoparticles made of Fe₂O₃ could be responsible for the presence of iron on the surface of the adsorbent.

FTIR analysis

The following table presents the findings of an FTIR analysis performed on calcium oxide (CaO) and a CaO/Fe₂O₃ nanocomposite both before and after the adsorption of Zn(II) ions. A prominent and wide peak was detected in the wavelength range of 1792 and 1838 cm⁻¹ for CaO that was generated from eggshell. This peak was an indication of vibration of functional groups of N-H and O-H that are present in the structure of CaO. Additionally, peaks in the wavelength ranges of 1332, 1509, 1553, and 2419 cm⁻¹ were identified, each

of which indicated that functional group vibrations of C = O and C-O-O were present, respectively. The presence of Ca-O in the structure of calcium oxide is the cause of the minute peak that can be found at 974 cm⁻¹. During the process of creating the CaO/Fe₂O₃ nanocomposite, some peaks in the CaO structure were also altered. These variations could be induced by the formation of Fe₂O₃ nanoparticles in the CaO structure. In addition, the steep peak that was seen in the region of 3887 cm⁻¹ could be attributable to Fe-O vibrations that were present in the composite structure. Recent research has revealed that interactions between functional groups and Zn(II) ions are responsible for the changes that took place in the CaO structure as a result of using CaO/Fe₂O₃ nanocomposite as the adsorbent. These interactions were observed in the CaO/Fe₂O₃ nanocomposite.

XRD analysis

The results of an XRD examination that was conducted to determine the crystalline phases of CaO and a composite of CaO and Fe₂O₃ are shown below for your perusal. According to the data, the fact that CaO/Fe₂O₃ exhibits a number of peaks at various angles indicates that both of the aforementioned substances have a crystalline structure. In the CaO/Fe₂O₃ composite structure, both the range and intensity of the peaks that were present in the CaO structure were significantly altered. Also, multiple peaks at 2θ of 11.64°, 20.77°, 29.19°, 29.22°, 35.81°, and 62.63° are detected in the structure of CaO/Fe₂O₃ composite. These peaks can be attributed to the crystalline phases of (1000), (587), (641), (36), and (355), respectively, and demonstrate that Fe₂O₃ is present in the structure of CaO/Fe₂O₃ composite. As a consequence of this, these peaks demonstrate that Fe₂O₃ was manufactured in an appropriate manner.

Optimization of adsorption parameters

The influence of pH on the adsorption process

Adsorption of Heavy Metal Ions pH of the Solution The pH of the solution is very important in the adsorption of heavy metal ions. The influence of pH (in the range of 2-8) on the adsorption of Zn(II) by CaO/Fe₂O₃ nanocomposite is depicted here using a graph. The adsorption effectiveness of zinc(II) increased considerably from 52.56 percent to 97.32 percent when the pH was between 2 and 6. Due to the fact that Zn(II) and hydrogen ion (H⁺) both attempted to adsorb onto the active sites of the CaO/Fe₂O₃ nanocomposite at low pHs, the adsorption efficiency was significantly reduced. Because free Zn(II) ions are present in the forms of ZnOH²⁺, Zn(OH)₄⁴⁺, and Zn₃(OH)₄⁵⁺ for adsorption on the negative side of the CaO/Fe₂O₃ nanocomposite, it was discovered that the adsorption of Zn(II) at a pH of 3-5 is favourable.

In addition, the adsorption efficiency and the slope of the plot both decreased for pH values that were larger than 6. This could be because of competition on the active sites of the CaO/Fe₂O₃ nanocomposite between the ion Zn(II) that results from precipitation and the hydroxyl ion that results from adsorption. As a consequence of this, a pH value of 6 was concluded to be optimal, and this value was utilised in subsequent research.

The influence that adsorbent dose has on the adsorption process

The efficacy and capacity of adsorption of Zn(II) ion on CaO/Fe₂O₃ nanocomposite at adsorbent dosage range of 0.1-0.5 g/L are shown in the figure. The adsorbent dosage range was 0.1-0.5 g/L. The graph makes it abundantly evident that increasing the amount of adsorbent used improves the efficiency of the adsorption process. In addition, the adsorption equilibrium was found to be achieved at a dosage of 0.1 g/L for the adsorbent. At an adsorbent dosage of 0.1 g/L, it was found that the maximum adsorption efficiency could be achieved, which was 88.53%. At adsorbent dosages higher than 0.1 g/L, there was no detectable improvement in the effectiveness of the adsorption process. It is possible that an increase in the availability of active sites is the cause of an increase in the adsorption efficiency that occurs when the dosage of the adsorbent is raised. In addition, as a consequence of

the saturation of available adsorption sites, the level of adsorption efficiency that was present at the point of equilibrium did not change. As the amount of adsorbent rose, the capacity for adsorption decreased. This result can be dictated by the interference and collision of adsorbent active sites at a higher adsorbent dosage, or it can be that the concentration of Zn(II) ion in solution is inadequate to establish bonds with accessible and active sites on the adsorbent surface of CaO/Fe₂O₃ nanocomposite. adsorbent active sites can interfere with each other and collide.

The influence of the starting concentration of zinc 2+ ions

We looked into how the beginning concentration of Zn(II) ions affected the amount of Zn(II) ions that could be adsorbed by a nanocomposite of CaO and Fe₂O₃. The graph shows that the adsorption effectiveness improved as the initial concentration of Zn(II) ion became higher. This result might be explained by an increase in the initial concentration of Zn(II) ion, which provides the necessary driving forces for mass transfer between the liquid and solid phases. It has been discovered that the ratio of accessible active sites to Zn(II) ions in the aqueous phase is larger when the concentration of the Zn(II) ions is lower. When the concentration was increased, however, the active site became saturated, which caused the ratio to decrease. As a consequence of this, the efficiency of the adsorption process was 17.73% when the starting concentration of Zn(II) was 50 mg/L.

The influence of contact duration on the adsorption process

We looked at how the amount of time that Zn(II) ions were in contact with the CaO/Fe₂O₃ nanocomposite affected the adsorption performance of Zn(II) ions by the nanocomposite. As can be seen in the figure, increasing the contact duration from 10 to 180 minutes resulted in Zn(II) adsorption by CaO/Fe₂O₃ nanocomposite occurring rapidly. After achieving equilibrium at 180 minutes, the rate of adsorption began to gradually decline. There is a possibility that the greater accessibility of active sites in the CaO/Fe₂O₃ nanocomposite is the cause of the quicker adsorption at initial contact times.

In addition, a drop in adsorption efficacy during the course of the contact time may be the result of saturation, which leads to a reduction in the number of active sites. In light of these data, it is reasonable to conclude that a contact time of one hundred and fifty minutes is adequate for Zn(II) ion adsorption under the present experimental conditions.

The influence of temperature on the process of adsorption

The influence of temperature on the efficiency of Zn(II) adsorption by CaO/Fe₂O₃ nanocomposite from aqueous solution is depicted in the graph. As already said. According to the figure, an increase in the adsorption efficiency from 93.87% to 96.73% was achieved by lowering the temperature from 310 to 300 k. This result may be explained by an increase in the reaction that occurs between the Zn(II) ion and the active sites of the CaO/Fe₂O₃ nanocomposite. This observation provides further evidence that the adsorption of Zn(II) ions to the CaO/Fe₂O₃ nanocomposite generates heat. In addition, it was demonstrated that the efficacy of adsorption decreased below 320 kelvin, which can be related to the instability of the Zn(II) ion and the formation of complexes at lower temperatures. The adsorption of Zn(II) on CaO/Fe₂O₃ nanocomposite at room temperature is the recommended method due to the favourable economics of this approach.

The isotherms of adsorption

The Isotherm of Langmuir

The Langmuir isotherm model for Zn(II) adsorption on CaO/Fe₂O₃ nanocomposite was utilized, and the findings are shown here. The concentrations used ranged from 10 to 80 mg/L. The value of the regression coefficient (R²) for Zn(II) was calculated to be 0.961. In addition, the KL value was determined to be in the range of 0.092, which, given the conditions described earlier, is appropriate. In addition, the CaO/Fe₂O₃ nanocomposite exhibited a maximum adsorption capacity of 17.857 mol g⁻¹. As a direct

consequence of this, the adsorption of Zn(II) ion using CaO/Fe₂O₃ nanocomposite adheres to the Langmuir isotherm model and takes place on the homogenous sites and monolayer surfaces of the CaO/Fe₂O₃ nanocomposite adsorbent.

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Freundlich Isotherm

The results of the Freundlich isotherm model as well as the isotherm model of Zn(II) ion adsorption onto CaO/Fe₂O₃ nanocomposite are provided below. In Zn(II) ion adsorption by CaO/Fe₂O₃ nanocomposite, the constant value of n was estimated to be 1.48, which indicates low adsorbent surface heterogeneity. In a similar manner, it was found that the determination coefficient (R^2) of the Zn(II) ion was 0.946.

Temkin Model

According to the figure, multilayer adsorption will take place when the adsorbent surface increases, the adsorption heat of all molecules decreases linearly, and adsorption is typified by uniform or maximum distribution binding energies. In a similar manner, it was found that the determination coefficient (R^2) of the Zn(II) ion was 0.994.

Adsorption Kinetics model

Pseudo first order kinetic model

The adsorption capacity as well as the estimated values of $K = 0.039$ and $q_e = 4.998$ are displayed in the image. The values of R^2 in this pseudo first order equation imply the least amount of elimination, and as a consequence, the pseudo first order cannot be satisfied by the data. As a consequence of this, this mechanism takes place during the process of physisorption.

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Pseudo-second-order model of the kinetic process

$$y = -0.017x + 0.698$$

$$R^2 = 0.975$$

9

Pseudo second order kinetic model

The adsorption capacity of CaO/Fe₂O₃ is presented by a graph with the values $K = 0.025$ and $q_e = 6.993$ displayed and calculated respectively. As a consequence of this, the values of R^2 in this equation of pseudo-second order indicate a greater amount of elimination, and their values correspond to the pseudo-second order. As a consequence of this, this mechanism takes place during the process of chemisorption.

$$y = 0.143x + 0.819$$

$$R^2 = 0.998$$

SUMMARY AND RESULTS OF THE STUDY

One of the most important issues facing the current generation is the provision of drinkable water made from very dirty industrial waste. It has been shown that treating wastewater with the suitable adsorbents can be quite successful while also being very cost-effective. The present review offered a summary of adsorption-based techniques for the elimination of pollutants. The purchasing and selling of goods and services through the use of digital networks is referred to as "electronic commerce."

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1

In this study, the removal of Zn(II) ions from aqueous solutions was carried out in a batch approach using CaO/Fe₂O₃ nanocomposite as a novel adsorbent. The method used was batch chromatography. The chemical

coprecipitation method was utilised in order to fabricate the nanocomposite in question. After that, the impact of a number of parameters, including pH level, beginning concentration of Zn(II) ion, contact time, and adsorbent dosage, was investigated. The pH range of 5 was shown to produce the greatest amount of adsorption yield. As the pH rose over this range, hydroxide ions began to precipitate out of solution, which resulted in a reduction in the efficiency of the treatment. In addition, the highest possible adsorption yield was found to be achieved by using a composite concentration of 2 g/L. Under these conditions, it was concluded that a removal efficacy of 97.53% was optimal. In addition, increasing the temperature over 30 degrees Celsius resulted in a reduction in adsorption effectiveness. This indicated that exothermic adsorption of Zn(II) ion on the adsorbent occurred, as well as damaged active sites on the surface of the CaO/Fe₂O₃ nanocomposite. In order to analyse the surface of the adsorbent, FTIR, XRD, EDAX, and FESEM were all utilised as investigative tools. The Langmuir isotherm model was discovered to be superior to the others in the isotherm study due to the fact that it had a greater correlation coefficient. (R^2). The maximum amount of adsorption that can occur per unit of weight, as calculated by the Langmuir model, is 60.60 mg/g. A quasi-second-order model was discovered through the study of the kinetics of Zn(II) ion adsorption onto CaO/Fe₂O₃ nanocomposite. The sorption thermodynamic was able to establish the spontaneous and exothermic sorption of Zn(II) ion by the CaO/Fe₂O₃ nanocomposite.

When it comes to the creation of materials, several methods have their own unique benefits and drawbacks, which influences the efficiency of the adsorbent in metal removal. As a consequence of this, several straightforward modifications and optimisations are essential in order to accomplish cost-effective and eco-friendly adsorbent development. The adsorption process is affected by a number of factors, including temperature, ionic strength, porosity of the material, surface area, affinitive functional group sites, adsorbate-adsorbent interaction, solution pH, and adsorbent dosage. In addition, adsorption isotherm and kinetic models are essential components for improved adsorption system design in addition to adsorption system tracking

in monolayer and multilayer adsorption systems. In an aquatic environment, traditional materials also have the ability to absorb metals. Regeneration and reuse of adsorbents may be possible if the minute size, surface area, shape, and magnetic properties of nanoparticles are maintained. When the nanocomposites have served their purpose, they can be recycled by first eliminating the heavy metals that were collected using solvents. It should come as no surprise that doing so would raise the total quantity of chemicals utilised in the procedure. To investigate the selectivity of the removal process, additional study is required to analyse the behaviour of nanocomposites when various heavy metals are present in water. This will allow the selectivity of the removal process to be investigated. In addition, adsorption experiments should be carried out not only on the metals that were investigated for this thesis but also on materials that are not heavy metals. In conclusion, several functionalizations could be examined in order to increase the efficiency of the removal process. It has been demonstrated that research into the synthesis, processing, characterisation, and application of nanomaterials for the removal of heavy metals from industrial applications like wastewater treatment or biological systems is likely to bear fruit.

There are numerous options that might be examined in the future to further investigate the improved performance of the materials contained in this thesis with the goal to provide an enhanced comprehension of wastewater treatment facilities. These pathways can be investigated further to further research improve the qualities of the materials provided in this thesis. It is suggested that various isotherms & kinetic models be sought for in order to conduct an in-depth study of the process of adsorption dynamics. The BET model and numerous other isotherms, which include the Langmuir & Freundlich isotherms, weren't taken into account in our research until recently. It is also critical to comprehend the impact of adsorption thermodynamics on our system. Businesses that treat wastewater must develop novel adsorbents which can be employed on a large scale, and that work must be done in close collaboration with university-affiliated research organisations. Given the discharge of these metals and their possible hazardous consequences on both individuals and the environment,, there is a pressing need to prioritise

the synthesis of innovative materials and the applications of those materials in the process of removing such metals. In the work that has been done recently, the focus has been placed on the batch mode, which is an approach that has proven to be successful when it comes to conducting laboratory investigations. Because this method does not accurately reflect applications in the real world, it is necessary to investigate different methodologies, such as constant, stationary, and packed bed reactor systems, in order to ascertain the effects of nanomaterials for adsorption at varying concentrations.

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