

**ADSORPTION APPROACH FOR EFFICIENT REMOVAL OF
Cr⁶⁺ FROM AQUEOUS SOLUTION USING Fe₃O₄/GRAPHENE
OXIDE NANO COMPOSITE**

Submitted in partial fulfilment of the requirement for the award of

Bachelor of Technology Degree in
Chemical Engineering

By

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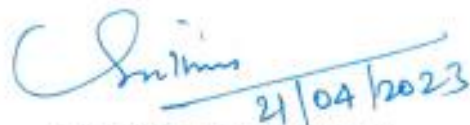
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BONAFIDE CERTIFICATE

This is to certify that this project report is the bonafide work of JAYASURIYA R (39190009) and RISHIKIRAN. A (39190027) who carried out the project entitled "ADSORPTION APPROACH FOR EFFICIENT REMOVAL OF CR⁶⁺ FROM AQUEOUS SOLUTION USING Fe₃O₄/GRAPHENE OXIDE NANO COMPOSITE" under our supervision from November 2022 to April 2023


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We Jayasuriya R (39190009) and Rishikiran A (39190027) hereby declare that the Project Report entitled "**ADSORPTION APPROACH FOR EFFICIENT REMOVAL OF Cr⁶⁺ FROM AQUEOUS SOLUTION USING Fe₃O₄/GRAPHENE OXIDE NANO COMPOSITE**" done by us under the guidance of Dr. M. Karthikeyan M.Tech, Ph.D., at SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY, CHENNAI 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 resources are under threat today more than ever, and many of them are polluted by anthropogenic sources such household trash, agricultural waste, and industrial waste. The primary goal of wastewater treatment is the elimination of the many contaminants found in wastewater, including pathogens, suspended particles, organic carbon, nutrients, inorganic salts, and heavy metals. As a result, before wastewater is discharged into natural water streams, it must still be treated. The ultimate goal of wastewater treatment is to offer protection for both the environment and human health. In this study we have demonstrated the removal of Cr^{6+} metal from aqueous solutions with the help of $\text{Fe}_3\text{O}_4/\text{GO}$ from graphite. This study contains the characterization results of Scanning Electron Microscopy (SEM), SEM with EDX, X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR). The operating condition such as pH, Contact time, Concentration, Dosage, Temperature were considered. Kinetic study and Adsorption Isotherm is calculated using the results obtained.

Keywords Chromium. Graphene oxide. Aqueous Solution

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LIST OF ABBREVIATIONS

AAS	Atomic Adsorption Spectrometer
BET	Brunauer-Emmett-Teller analysis
Cr	Chromium
EPC	Environmental Protection Agency
SEM	Scanning Electron Microscopy
FT-IR	Fourier Transform Infrared
GO	Graphene Oxide
Mg/L	Milligram per litre
UV	UltraViolet
XRD	X-Ray Diffraction

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CHAPTER. 1

INTRODUCTION

1.1. BACKGROUND AND CONTEXT

A collection of metallic elements with a relatively high density and atomic weight are known as heavy metals. In industrial operations including mining, smelting, and metal refining, they are often utilised. But if released into the environment, they might have a negative impact on both the ecosystem and human health owing to their poisonous nature.

A heavy metal is a metallic element having a relatively large atomic weight, specific gravity, or density. In industrial waste water, heavy metals including lead, cadmium, mercury, cobalt, and others could be present. It is vital to remove heavy metals from waste water since they do not naturally degrade and are very toxic to aquatic life even in little amounts. Adsorption is a mass-transfer technique in which the surface of the solid receives the deposition of the dissolved solid from the liquid as a result of physical or chemical contact. When dissolved particles from a liquid attach to the surface of a solid, the process is known as adsorption.

The use of inexpensive adsorbents made from industrial wastes as an alternative to pricey traditional methods of collecting heavy metal ions from wastewater is of great interest to many researchers. Fly ash, waste slurry, red mud, lignin, sugar beetroot pulp, blast furnace slag, tea industry waste and sugar cane bagasse are examples of promising materials. Utilising two different Turkish fly ashes nickel [Ni^{2+} , Cu^{2+} , and Zn^{2+}] were removed from an aqueous solution. It was discovered that fly ash, which has a high calcium concentration, works just as well as activated carbon as a metal adsorbent. In this study, the effects of four variables—fly ash/lime mass ratio, fly ash/lime absorbent type, solution temperature, and sorbent concentration—were examined in relation to the removal of the five metallic ions (Cu^{2+} , Ni^{2+} , Zn^{2+} , Cd^{2+} , and Pb^{2+}) [1]. With a solution temperature of 66 C and a sorbent ratio of 0.66, they found that an adsorbent with a concentration of 122 g L⁻¹, or fly ash and 5% by mass of lime, produced both high adsorption and low dissolution. Namasivayam tested the effectiveness of eliminating hazardous anions, dyes, heavy metals, and organic compounds from water using jatropha husk activated carbon generated from agro-industrial solid waste.

According to studies, 97% of nickel and 61% of the original percentage of chromium IV could both be removed. Several studies have been conducted on the removal of heavy metals from waste water by industrial waste. the efficiency of removing certain types of industrial waste. The investigation into utilising agricultural leftovers as adsorbents to remove heavy metals from industrial wastewater has recently attracted a lot of attention. The method of employing agricultural wastes in the bioremediation of heavy metal ions is known as bio-sorption.

Certain biomass may passively concentrate and quell contaminants onto its cellular structure via to a physiochemical process known as biosorption. Many scientists believe that this phenomena will provide a practical means of removing dangerous heavy metals from industrial wastewater and will aid in environmental cleaning. They looked into and compared the ability of pectin-rich fruit peels to absorb cadmium metal and found that grape skin had the highest capacity when compared to orange, lemon, apple, etc. The majority of heavy metals, including Pb^{2+} , Cd^{2+} , Zn^{2+} , Cu^{2+} , and Ni^{2+} , have higher sorption capacities for rice hull than saw dust because of the silica functional group on rice hull and its high surface area, according to a study and comparison of the heavy metal removal capacities of rice hull and saw dust.

The biosorption capabilities of raw ruin and ruin resin were 26.3 and 41.6 mg/g, respectively, according to biosorption and desorption studies on Cr^{6+} using innovative ruin and ruin resin bio adsorbents. The major functional groups linked to the resin for the aim of eliminating chromium from waste water were the hydroxyl, carboxyl, and carbonyl groups. It was suggested that toxic heavy metal ions could be removed from aqueous solutions using chemically altered agricultural waste (okra biomass). It was discovered that the okra biomass had a very high capacity for adsorption of Pb^{2+} , Cd^{2+} , Zn^{2+} , and Cu^{2+} at respective concentrations of 273.97 mg/g, 121.51 mg/g, and 57.11 mg/g. Seaweed *Ascophyllum nodosum*, sawdust, and reed plant root *Phragmites australis* were three low-cost bio adsorbents that were contrasted. Of the three, sawdust exhibited the most potential for lead adsorption, whereas seaweed showed the best cadmium adsorption capacity. Which heavy metals can be removed through biosorption depends on the functional group attached to the bio adsorbents; for instance, the silica group has a stronger affinity

for copper, zinc, and lead than the hydroxyl group does for copper, lead, and nitrogen.

When selecting an adsorbent to treat wastewater, two key features of this technology; Technical applicability and economic effectiveness should be taken into consideration. Adsorbents may be classified as being made of natural materials, man-made materials, agricultural waste, and biological waste. Materials made of nanoparticles mixed with a polymer matrix or other materials are known as nanocomposites. Large surface area, excellent mechanical strength, and changeable surface chemistry are just a few of their special qualities. These characteristics make them perfect for a variety of uses, including water filtration, medication delivery, and catalysis.

The adsorption procedure is one of the most popular ways to remove heavy metals from water. In this process, heavy metal ions are bound to an adsorbent material's surface. Activated carbon, zeolites, and many kinds of nanoparticles are examples of adsorption materials that may be either natural or artificial. Nanocomposites have been thoroughly studied as potential adsorbent materials for heavy metal removal. They have a large surface area and a diverse surface chemistry, which may allow them to successfully adsorb heavy metal ions from water. Its properties may also be easily changed in order to improve its capacity to adsorb and select certain heavy metal ions [2].

One of the most common sources of heavy metal pollution, which is a severe environmental problem, is industrial effluent. Various industrial procedures, such as mining, electroplating, and metal processing, have the potential to discharge significant amounts of heavy metals into the environment. These procedures may also contaminate water sources and harm both human and ecological health.

Adsorption is the method used most often to remove heavy metals from wastewater. Heavy metal ions may be physically or chemically removed from water by the process of adsorption, which involves binding the metal ions to the surface of an adsorbent material. Traditional adsorbents such activated carbon and zeolites have been used for this purpose, but they have limitations including poor selectivity, low adsorption capacity, and high cost.

Nano-adsorbents composed of nanoscale particles or fibers were investigated as an alternative to conventional adsorbents for the removal of heavy metals from wastewater. These materials have many advantages over traditional adsorbents, including a wide surface area, changeable surface chemistry, and strong selectivity to certain heavy metal ions. Nano adsorbents may be made from a wide range of materials, such as metal oxides, polymers, and carbon nanotubes.

Recent years have seen a lot of study on the use of nano adsorbents to remove heavy metals from wastewater. According to studies, these materials may efficiently and selectively remove heavy metals from wastewater, including lead, cadmium, and mercury. Furthermore, compared to conventional adsorbents, the usage of nano adsorbents may lead to reduced prices and quicker removal times. However, using nano adsorbents to remove heavy metals from wastewater comes with a number of difficulties. The potential toxicity of these compounds is one of the main obstacles. According to certain research, releasing certain kinds of nano adsorbents into the environment might be harmful to human health. It is crucial to guarantee the appropriate handling and disposal of these items.

The expansion of laboratory technologies to an industrial scale presents another difficulty. Nano adsorbent synthesis and manufacture may be difficult, requiring specialized knowledge and tools. Therefore, for the widespread use of nano adsorbents in industrial applications, the development of affordable and scalable manufacturing procedures is crucial. Despite these challenges, there are some advantages to employing nano adsorbents rather than traditional adsorbents to remove heavy metals from wastewater. They offer high selectivity, efficiency, and adsorption capacity and are easily tailored to specific applications. Since they may also shorten the cost and time of the heavy metal removal procedure, they are a promising option for treating industrial wastewater.

Using adsorption methods to remove heavy metals from nanocomposites is a key area of research with the aim of developing efficient and economical methods for removing heavy metals from water. This area of research has a big impact on the environment and people's health, and it has the potential to enhance the development of environmentally friendly and sustainable technologies.

In conclusion, the research of employing nano adsorbents to remove heavy metals from wastewater is an area that is rapidly increasing and has tremendous potential to have a good impact on both the environment and the economy. Although there are drawbacks to this approach, further investigation and development may lead to the widespread use of nano adsorbents as a cost- and sustainably-effective method of reducing heavy metal pollution [3].

During the adsorption procedure, pollutants are removed from a solution by binding contaminants to the surface of $\text{Fe}_3\text{O}_4/\text{GO}$ nanoparticles. $\text{Fe}_3\text{O}_4/\text{GO}$ has unique qualities, such as a large surface area, magnetic capabilities, and chemical stability, which make it a viable adsorbent for a variety of applications. The adsorption process in $\text{Fe}_3\text{O}_4/\text{GO}$ is predominantly driven by the physical and chemical interactions between the molecules of the adsorbate and the adsorbent surface. Examples of physical interactions include van der Waals forces, hydrogen bonds, and electrostatic forces, while chemical interactions include covalent or ion-dipole bonds.

The ability of $\text{Fe}_3\text{O}_4/\text{GO}$ to adsorb is influenced by a variety of variables, including the concentration of the adsorbate, pH, temperature, and contact time. $\text{Fe}_3\text{O}_4/\text{GO}$'s adsorption capacity may be increased by optimising these variables as well as by functionalizing or doping the adsorbent's surface.

$\text{Fe}_3\text{O}_4/\text{GO}$ has undergone considerable research for the removal of numerous impurities from water, including heavy metals, organic pollutants, and pigments. The adsorption of heavy metals onto $\text{Fe}_3\text{O}_4/\text{GO}$ requires the establishment of coordination bonds between the metal ions and the functional groups on the surface of the adsorbent. $\text{Fe}_3\text{O}_4/\text{GO}$ has a great potential for adsorbing heavy metals including lead, cadmium, and mercury, as has been shown.

Adsorption of organic pollutants onto $\text{Fe}_3\text{O}_4/\text{GO}$ includes physical adsorption of the contaminants onto the surface of the adsorbent. The ability of $\text{Fe}_3\text{O}_4/\text{GO}$ to effectively adsorb a number of organic pollutants, including as dyes, phenols, and polycyclic aromatic hydrocarbons (PAHs), has been shown.

In conclusion, $\text{Fe}_3\text{O}_4/\text{GO}$ is an appropriate adsorbent for the removal of pollutants from water because of its enormous surface area, magnetic characteristics, and chemical stability. $\text{Fe}_3\text{O}_4/\text{GO}$ characteristics and synthesis must be enhanced for

certain applications, and further study is required to see if it can remove other pollutants from water.

Fe₃O₄/GO nanocomposites are a kind of hybrid material that has drawn a lot of attention in the field of nanotechnology due to its peculiar blend of magnetic and electrical properties. While graphene oxide alone has high mechanical and electrical conductivity, Fe₃O₄ nanoparticles in the matrix provide the material stronger magnetic properties. Graphene oxide is commonly reduced in the presence of Fe₃O₄ to produce Fe₃O₄/GO nanocomposites. The finished item has unique properties that make it desirable for a variety of applications, including biomedical imaging, medicine delivery, and environmental remediation.

Biomedical imaging is one of Fe₃O₄ nanocomposites' most important uses. The graphene oxide matrix may be functionalized with targeted molecules to increase imaging specificity, and Fe₃O₄/GO nanoparticles can be used as contrast agents for MRI due to their magnetic characteristics. Additionally, Fe₃O₄/GO nanocomposites have significant promise as drug delivery methods. Utilizing magnetic fields, Fe₃O₄ nanoparticles may be utilized to direct nanocomposites to certain body locations, while functionalizing the graphene oxide matrix can improve drug loading and release.

Fe₃O₄/GO nanocomposites have also been investigated for their potential in environmental remediation. While the graphene oxide matrix may be utilised to absorb pollutants and contaminants, the magnetic characteristics of Fe₃O₄/GO nanoparticles can be employed to separate nanocomposites from contaminated water. Energy storage is another possible use for graphene oxide- Fe₃O₄ nanocomposites. Graphene oxide has a high electrical conductivity and a big surface area, making it a great material for supercapacitors. The nanocomposite material created by adding Fe₃O₄ to the graphene oxide matrix exhibits enhanced capacity and cycle stability, making it a strong contender for high-performance energy storage devices [4].

Additionally, Fe₃O₄/GO has been researched for its potential in catalysis. The graphene oxide matrix serves as a stable and conductive substrate for the Fe₃O₄ particles, which may function as effective catalysts for a variety of chemical processes. Advances in nanotechnology and materials science have made it easier

to create Fe₃O₄/GO nanocomposites. The production of nanocomposites with customized characteristics for particular purposes has been made possible by the ability to regulate the size, shape, and distribution of the nanoparticles inside the graphene oxide matrix. Fe₃O₄/GO nanocomposites, in general, provide a special mix of characteristics that appeal to a wide range of applications. Although more study is required to completely comprehend their potential qualities and uses, they have the potential to enhance our everyday lives in a number of ways. Bio adsorbents, low-cost adsorbents, and new adsorbents have been the most frequently studied adsorbents in recent years for the treatment of wastewater contaminated with heavy metals; however, other than modified natural adsorbents, no adsorbents have been used industrially to remove heavy metals. The approach is not in any way scaled up or made available to the industry. The study's only goal has been to determine whether or not that substance is suitable for getting rid of heavy metals and, if so, how successful it is at doing so. There hasn't been a thorough examination of the desorption process and disposal of the adsorbent utilised to extract heavy metals. Since many of them have stated, although pre-treatment and expensive handling are still required for the disposal of sludge, adsorbents like industrial waste, agricultural waste, and biological waste all have excellent adsorption capacity for the removal of heavy metals and are also readily available.

Further study is required to lower the cost of pre-treatment, regeneration, and disposal. The primary field of research for the removal of heavy metals is without a doubt the utilisation of readily available adsorbents.

1.2. STATEMENT OF THE PROBLEM

Heavy metals in industrial waste water pose a serious threat to the environment, and their removal is crucial to avoid pollution and safeguard public health. Precipitation, ion exchange, and filtration through membranes are examples of traditional treatment techniques, although they have drawbacks such high cost, poor efficiency, and limited scalability. Therefore, a low-cost and efficient method of extracting heavy metals from industrial effluents is required.

Adsorption is a potential method for removing heavy metals because of its great effectiveness, cheap cost, and simplicity of use. Due to their substantial surface area

and strong magnetic characteristics, $\text{Fe}_3\text{O}_4/\text{GO}$ nanocomposites have shown remarkable heavy metal adsorption abilities [5]. However, the effectiveness of these nanocomposites for the removal of heavy metals from industrial wastewater has not yet been completely investigated, and in order to maximise efficiency and scalability, it is necessary to optimise their synthesis and working conditions.

The goal of this work is to evaluate the efficiency of $\text{Fe}_3\text{O}_4/\text{GO}$ nanocomposites for the adsorption-based removal of heavy metals from industrial effluents as well as to maximise the synthesis and operating conditions for scalability and maximum efficiency. This work covers other significant aspects impacting heavy metal adsorption by graphene oxide- Fe_3O_4 nanocomposites in addition to optimising the production and operation conditions. The pH of the solution, the initial concentration of heavy metals, the amount of time the nanocomposites have to come into contact with the effluent, and the existence of other competing ions are some of these variables.

A series of tests will be performed as part of the research to assess the effectiveness and adsorption capacity of graphene oxide- Fe_3O_4 nanocomposites for different heavy metals, including Cr^{6+} . To identify the ideal circumstances for maximal adsorption, the effect of several factors, including pH and starting concentration, is researched. $\text{Fe}_3\text{O}_4/\text{GO}$ nanocomposites may be used to remove heavy metals from industrial effluent, according to the findings of this research. The creation of an affordable and effective adsorption method that can be used on a wide scale is made possible by the optimised chemistry and operating conditions. As a result, the issue of heavy metal pollution in industrial effluents will be addressed and a greener, more sustainable approach to wastewater treatment will be developed [6].

1.3. OBJECTIVES OF THE STUDY

This study's objective is to determine how well a nanocomposite adsorbent removes heavy metals from wastewater with an emphasis on the material's capacity, selectivity, and reusability. To establish if the nanocomposite has the potential to be a workable solution for the removal of heavy metal from wastewater, the objective is to optimise the process parameters and assess the adsorption kinetics and equilibrium isotherms.

The study's other goal is to pinpoint the critical variables, such as pH, contact duration, temperature, starting heavy metal concentration, and adsorbent dose, that impact the nanocomposite's ability to adsorb. The effectiveness of the nanocomposite is evaluated in comparison to other adsorbents often used for the removal of heavy metals from wastewater.

Additionally, the research will use analytical methods including X-ray diffraction (XRD), Fourier-transformed infrared spectroscopy (FTIR), and Scanning electron microscopy (SEM) to look into the adsorption process of heavy metals on the nanocomposite surface. The manufacture and characterization: To make GO, one must first synthesise it. Then, using a variety of analytical methods, such as X-ray diffraction, Fourier transform infrared spectroscopy, transmission electron microscopy, atomic force microscopy, and Raman spectroscopy, one must characterise GO's physicochemical characteristics, including its structure, size, morphology, and surface chemistry.

Functionalization is the process of altering the surface of GO by functionalizing it with different chemical groups or biomolecules in order to improve its properties or tailor its surface for specific applications [7]. Covalent or non-covalent functionalization may be involved, and the study might examine how functionalization affects GO properties.

Uses: Investigate the potential applications of GO in a variety of fields, including as catalysis, energy storage, sensing, biological applications, and environmental cleanup. The study may include analysing how GO-based materials function in certain applications and determining how effective and efficient they are.

Analysis of the toxicity and environmental effects of GO and its derivatives in a variety of contexts, including in vivo and in vitro research as well as environmental fate and transport investigations. This kind of study aims to identify potential problems associated with the use and disposal of GO and to provide solutions to mitigate such risks.

The outcomes of this research are anticipated to provide useful knowledge on the usage of nanocomposites as an efficient and long-lasting technique for heavy metal removal from wastewater. The findings of this research may be utilised to build an efficient and affordable heavy metals treatment procedure for industrial wastewater.

The research will also assess if it is feasible to regenerate and reuse the nanocomposite adsorbent, which may lower the cost of the treatment procedure overall and lessen its environmental effect.

This study's overarching goal is to create an effective and sustainable process for removing Cr^{6+} from wastewater using a nanocomposite adsorbent. This may assist preserve the public's health and address rising concerns about environmental heavy metal contamination [8]. The findings of this research may be utilised to enhance the development of environmentally friendly wastewater treatment systems as well as to encourage the use of nanotechnology in environmental applications.

1.4. SIGNIFICANCE AND POTENTIAL CONTRIBUTIONS OF THE STUDY

The potential contributions that this work might bring to the fields of wastewater treatment and environmental protection are what give it its significance. Due to their toxicity and endurance in the environment, heavy metals are a major environmental issue, and their presence in wastewater may have negative effects on ecosystem and human health. Therefore, it is crucial to create sustainable and efficient removal techniques.

This work may help shed light on the efficiency and viability of the promising strategy of using nanocomposite adsorbents to extract heavy metals from wastewater. The research might aid in the creation of a heavier metal-contaminated wastewater treatment method that is more successful and affordable, especially in industrial settings.

Engineers, scientists, and decision-makers may utilise the findings of this study to optimise the use of nanocomposite adsorbents in wastewater treatment and encourage the use of ecologically benign, long-lasting heavy metal removal methods [9]. The research may also help with the creation of rules and guidelines to deal with heavy metal contamination in the environment, which will eventually preserve the health of people and ecosystems.

Overall, the results of this research might have a big impact on environmental engineering and help to create a more sustainable and healthier environment for everyone. The research may also help to advance the use of nanotechnology in environmental applications. Large surface area, strong reactivity, and excellent

selectivity are just a few of the distinctive characteristics that nanocomposite adsorbents have that make them very efficient in adsorbing heavy metals from wastewater. This work may provide important insights into the usage of nanotechnology in environmental applications by examining the mechanism of heavy metal adsorption on the nanocomposite surface.

The research may also aid in the creation of a circular economy. The total cost of the treatment procedure may be decreased, and the environmental effect of the adsorption process can be reduced, by regeneration and reuse of the nanocomposite adsorbent. This may support the idea of a circular economy, which minimises waste and uses waste materials as resources.

Finally, the course may help future generations of engineers and scientists gain knowledge and skills in the area of environmental technology. The findings of this study may be utilised to create fresh, creative methods for removing heavy metals from wastewater, which may inspire more study and invention in this area.

In conclusion, this work has a wide range of importance and possible contributions. The research may help the development of sustainable and green technologies and contribute to a healthier and more sustainable environment by providing useful information on the usage of nanocomposite adsorbents to remove heavy metals from wastewater [10].

CHAPTER.2

LITERATURE SURVEY

A severe environmental issue, heavy metal contamination may impair both the ecology and people's health. As a result, several techniques, such as adsorption procedures, have been developed to remove heavy metals from wastewater. Nano-composites have been a popular adsorbent for heavy metal removal in recent years because of its large surface area, cheap cost, and customizable characteristics.

- **Chakraborty et al. (2020)** created a chitosan/multi-walled carbon nanotube composite. The findings demonstrated that the composite could remove up to 98% of lead ions from wastewater and had a high adsorption capability.
- **Ates et al. (2019)** created a nano-composite for the removal of cadmium ions from wastewater based on graphene oxide and magnetic nanoparticles in a different research. The results showed that the composite could remove up to 95% of cadmium ions from wastewater and had a high adsorption capability.
- **Yadav et al. (2020)** created a composite for the removal of nickel ions from wastewater that is based on activated carbon and zinc oxide nanoparticles. According to the findings, the composite exhibited a strong capacity for adsorption and could filter up to 96% of nickel ions from wastewater.
- **Hasan et al. (2020)** utilised a composite based on activated carbon and titanium dioxide to remove chromium ions from wastewater, while **Njoku et al. (2020)** used a composite based on chitosan and activated carbon to remove copper ions from wastewater.
- For the removal of arsenic from wastewater, **Wang et al. (2020)** created a new adsorbent made of magnetic iron oxide nanoparticles and altered graphene oxide. The results demonstrated that the adsorbent had a high capacity for adsorption and could eliminate up to 99.9% of arsenic from wastewater.
- To remove cadmium (Cd) from wastewater, **Zhang et al. (2020)** created a nanocomposite of manganese oxide and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cd ions, and that the pH and starting Cd concentration had an impact on the removal effectiveness.
- A composite material based on graphene oxide and manganese dioxide was created in a research by **Li et al. (2019)** to remove lead ions from wastewater. According to the findings, the composite could remove up to 95% of lead ions from wastewater and had a high adsorption capability.
- To remove lead (Pb) from wastewater, **Wu et al. (2019)** created a nanocomposite of reduced graphene oxide and iron oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Pb ions, and that the pH, contact duration, and starting Pb concentration had an impact on the removal efficiency.
- The removal of cadmium (Cd) from wastewater was examined by **Chen et al. (2021)** using a nanocomposite of chitosan and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cd ions, and that the pH, contact duration, and starting Cd concentration had an impact on the removal efficiency.

- To remove lead (Pb) from wastewater, **Keshavarz et al. (2020)** created a nanocomposite of iron oxide and activated carbon. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Pb ions, and that the pH and starting Pb concentration had an impact on the removal efficiency.
- In order to remove nickel (Ni) from wastewater, **Kumar et al. (2018)** created a manganese dioxide and graphene oxide nanocomposite. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Ni ions, and that the pH, contact duration, and starting Ni concentration had an impact on the removal efficiency.
- To remove chromium (Cr) from wastewater, **Deng et al. (2017)** created a nanocomposite using titanium dioxide and activated carbon. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cr ions, and that the pH, contact duration, and starting Cr concentration had an impact on the removal efficiency.
- Aluminium oxide and carbon nanotubes were combined to create a nanocomposite by **Gao et al. (2017)** for the removal of arsenic (As) from wastewater. The findings demonstrated that the nanocomposite had a high capacity for adsorbing As ions, and that the pH, contact duration, and starting As concentration had an impact on the removal efficiency.
- Magnetic carbon nanotubes and chitosan were combined to create a nanocomposite that **Li et al. (2021)** used to remove mercury (Hg) from wastewater. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Hg ions, and that the pH and starting Hg content had an impact on the removal efficiency.
- To remove cadmium (Cd) from wastewater, **Shao et al. (2020)** created a nanocomposite of zinc oxide and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cd ions, and that the pH and starting Cd concentration had an impact on the removal effectiveness.
- Similar to this, **Wang et al. (2021)** created a composite for the removal of copper ions from wastewater that is based on magnetic graphene oxide and Fe₃O₄ nanoparticles. The findings demonstrated that the composite could remove up to 98% of copper ions from wastewater and had a high adsorption capability.
- Using iron oxide and montmorillonite, **Wang et al. (2018)** created a nanocomposite for the removal of copper (Cu) from wastewater. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cu ions, and that the pH and starting Cu concentration had an impact on the removal efficiency.
- To remove lead (Pb) from wastewater, **Nair et al. (2017)** created a nanocomposite of polyaniline and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Pb ions, and that the pH and starting Pb concentration had an impact on the removal efficiency.
- Additionally, **Chen et al. (2018)** developed a composite material based on graphene oxide and hydroxyapatite to remove lead ions from wastewater.

CHAPTER.3

AIM AND SCOPE

3.1 AIM

This work intends to test the performance of a newly synthesized nanocomposite material for the adsorption-based removal of heavy metals from wastewater. In order to discover whether the nanocomposite material is effective in removing heavy metals from wastewater, batch adsorption studies will be utilised to assess the material's physical and chemical features. The study also attempts to compare the effectiveness of the nanocomposite material with other conventional adsorbents for heavy metal removal and to improve the adsorption process by adjusting parameters like pH, contact length, and adsorbent dosage.

The study will also examine the reusability and stability of the nanocomposite material for repeated adsorption cycles, create a theoretical model to explain the adsorption process and establish the kinetic and thermodynamic parameters, and show that the nanocomposite material can actually be used to remove heavy metals from real wastewater samples. The research will also shed light on the process through which heavy metals adhere to the nanocomposite material.

3.2 SCOPE

The goal of this research project is to examine the use of a special nanocomposite material for the adsorption-based removal of heavy metals from wastewater. The research will concentrate on determining the ideal circumstances for using the nanocomposite material in wastewater heavy metal removal, how different factors affect the efficiency of the adsorption process, and how well the nanocomposite material removes various kinds of heavy metals from wastewater. The material's stability and reusability will be evaluated via a number of adsorption cycles. A variety of analytical approaches will be used in surface characterisation tests to further our knowledge of how heavy metals interact with the nanocomposite material. The findings of this work will have practical consequences for developing solutions for treating wastewater that contains heavy metals, a significant problem in many businesses, that are both ecologically sound and economically viable.

CHAPTER.4

MATERIALS AND METHODS

4.1 MATERIALS

Hydrogen peroxide (H_2O_2), Sodium nitrate (NaNO_3), Potassium permanganate (KMnO_4), Sulphuric acid (H_2SO_4), expandable graphite flakes (acid washed), $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, NaOH, and hydrochloric acid (HCl) were purchased. The pH of the utilised solution was altered by adulting H_2SO_4 or NaOH solutions in the required amounts. The concentration of the solutions was determined using a UV-visible spectrophotometer. For this experiment, only materials of the highest quality were used. Deionized water was used throughout the experiment [11].

4.2 GRAPHENE-OXIDE SYNTHESIS

Graphite flakes were employed as the starting material in a modified Hummers method to create GO nanoparticles. The right synthesis techniques and level of oxidation determine the structure and characteristics of GO. 2g of sodium nitrate and 2g of graphite flakes were mixed for 4 hours in 90 mL of H_2SO_4 (Fig:4.1). This solution was gradually given 12 g of KMnO_4 while the temperature was maintained below 10°C . After another hour of stirring, the resultant liquid was progressively diluted. (184 mL). The next two hours were spent stirring this mixture at 35 to 40°C until it became pasty brown. After mixing, the mixture was held at 25°C for two hours. By adding 200 mL of fresh water and 48% of the H_2O_2 solution (40 mL), the reaction was stopped. Using deionized water and a 10% HCl aqueous solution, the mixture was rinsed until the pH of the final solution was neutral. The mixture was then sonicated after that. To create GO nanoparticles, the precipitate is dried for 24 hours at 60°C [12].



Fig.4.1: Experimental image of rotary shaker



Fig.4.2: Graphite flakes

4.3 Fe₃O₄/GO MAGNETIC NANOCOMPOSITE SYNTHESIS

The Nano-composite Fe₃O₄/GO was created by following a precipitation method. The synthesis of the composite was started by adding a known amount of GO (0.1 g) and FeSO₄·7H₂O (2.78 g) into 100 mL of Distilled water and ultrasonically treated the mixture for 30 min to receive a unique dispersion of Fe₃O₄/GO. The final pH = 11.0 of the solution was achieved by adjusting with 25% ammonium hydroxide. Then, the solution was magnetically stirred in a water bath at 95 °C for 4 h to obtain a Fe₃O₄/GO nanocomposite. The obtained Fe₃O₄/GO nanocomposite was washed two times with Distilled water and dried using the lyophilization method for further subsequent experiments.



Fig.4.3: Experimental image of magnetic stirrer



Fig.4.4: Experimental image of sonicator

4.4 PREPARATION OF STOCK SOLUTION FOR BATCH STUDY

100 mg/L stock solutions of the Cr^{6+} metal were produced. Additionally, working standard solutions were created every day from the stock solutions by appropriate dilution with deionized water. Adsorption tests were routinely carried out in batch manner. A magnetic stirrer was used to mix the adsorbent and 100 ml of the stock solution in a 500 ml beaker. At room temperature, the stirring rate was held constant at 250 rpm and the working temperature was set at 25°C. The pH of the solution was changed by adding HCl or an alkali solution, and the new pH was then measured using a pH metre. Using filter paper, the adsorbent nanoparticles were removed from the solution [14]. The absorbance of the test sample was monitored over several time periods to look for variations. The metal concentration in this study was determined using an ultraviolet-visible (UV-Vis) spectrophotometer. You can determine the adsorption capacity by using the **Eqn (1,2)**

$$q_e = [(C_0 - C_e)/V] M \quad (1)$$

$$\% \text{ Adsorption} = (C_i - C_e)/C_i \quad (2)$$

q_e amount of metal adsorbed in mg per gram of adsorbent.

C₀ and **C_e** (mg/L) are initial and equilibrium metal concentrations.

C_i and **C_e** are initial and equilibrium concentration of C^{6+} (mg/L), respectively.

V is the volume of solution.

M is the mass of adsorbent.

The Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FT-IR), X-ray Diffraction (XRD), and Brunauer-Emmett-Teller analysis (BET) were only a few of the analytical techniques used.

4.5 ISOTHERMS, KINETICS AND THERMODYNAMIC STUDY

The data from Cr^{6+} adsorption equilibrium was fitted using the Freundlich, Langmuir, and Temkin model.. We used linear forms of pseudo-first-order, pseudo-second-order, and intraparticle diffusions to estimate the rate of Cr^{6+} adsorption onto $\text{Fe}_3\text{O}_4/\text{GO}$ [15]. Entropy, enthalpy, and free energy calculations were used to study the kinetics of Cr^{6+} adsorption.

CHAPTER.5

RESULTS AND DISCUSSION

5.1 CHARACTERIZATION OF GRAPHENE OXIDE

5.1.1. Scanning electron microscopy (SEM)

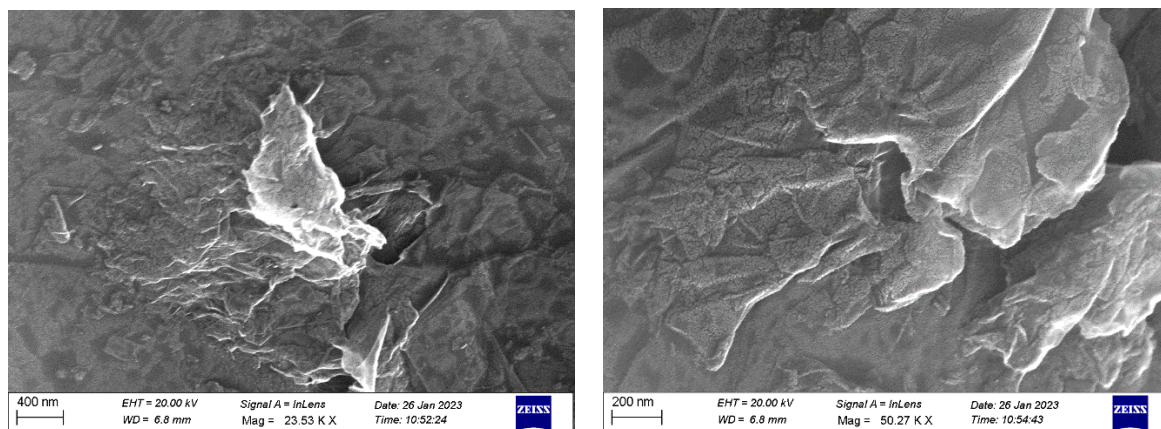


Fig.5.1, 5.2: Scanning electron microscopy image of GO

Scanning electron microscopy (SEM) analysis is powerful imaging and analytical technique that employs a focused electron beam to examine the surface morphology, elemental composition, and other properties of a sample with exceptional resolution. SEM analysis has wide-ranging applications in various fields, including materials science, nanotechnology, biology, geology, and forensic science, among others, owing to its versatility and precision in characterizing different types of samples [16]. Graphene oxide's scanning electron micrograph (Fig:5.1) reveals an extremely uneven and heterogamous surface structure. However, graphene has a layer structure at its core. When fictitiously modelled, GO displays a three-dimensional network at random aligned sheet-like structures with a stretched texture and centralized pore with a wide size distribution, which may be the best locations for heavy metal binding. forming a porous structure with a loose sponge-like shape that indicates the oxidation process at graphene has occurred. The (Fig:5.2) shows in the below is a scanned image at 200nm.

5.1.2 X-ray diffraction (XRD) study

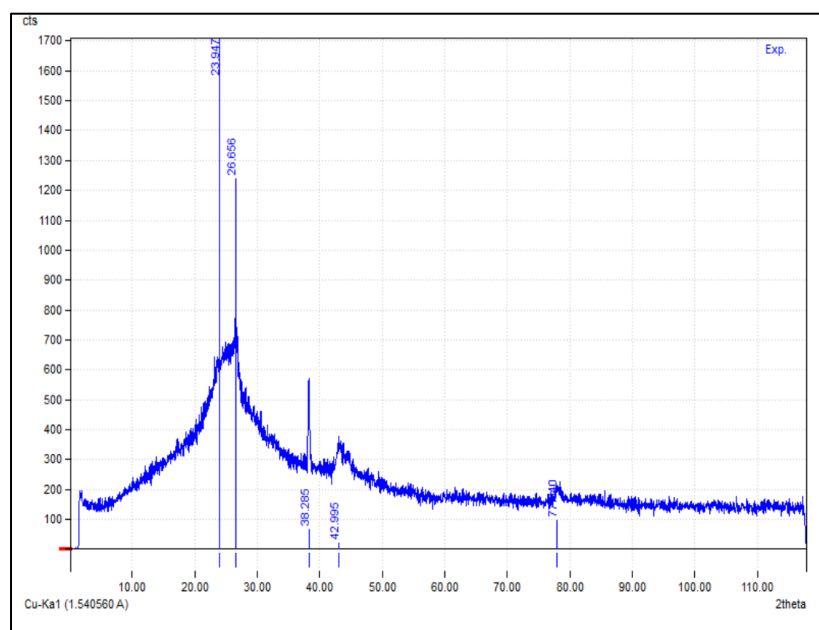


Fig.5.3: X-ray Diffraction

X-ray diffraction (XRD) analysis is a non-destructive and highly informative technique that employs X-ray beams to investigate the crystal structure, phase composition, and other properties of materials. Due to its ability to provide valuable insights into the atomic and molecular arrangement of materials, XRD analysis finds widespread applications in various fields, including materials science, solid-state physics, chemistry, geology, and pharmaceuticals. When applied to graphene oxide, XRD analysis can yield crucial information about the interlayer spacing, crystallite size, and stacking arrangement of graphene oxide layers, which are key to understanding its structural properties. The interlayer spacing can be determined from the position of the diffraction peak, providing insights into the degree of oxidation and the presence of the functional groups on the graphene oxide layers [17]. Graphene oxide's XRD pattern is seen in (Fig:5.3), respectively. A single broad peak at $2\theta=23.9$, which corresponds to an intermediate layer distance of 0.83 nm, dominated the XRD pattern GO. The peak at $2\theta = 38.2$ disappearing indicates that the product has undergone chemical oxidation and is fully oxidised. The earlier studies found almost identical findings.

5.1.3 Fourier Transform Infrared Spectroscopy (FT-IR).

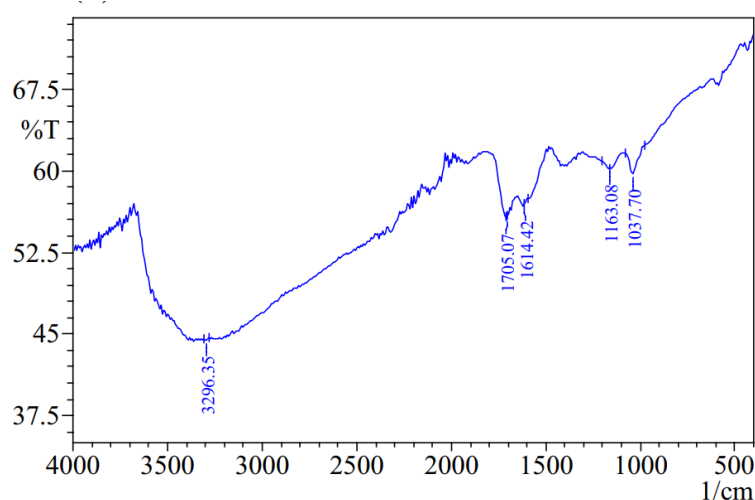


Fig.5.4: FT-IR of graphene oxide

Fourier Transform Infrared (FT-IR) analysis is a well-established and trustworthy technique for analysing the molecular composition, functional groups, and chemical properties of materials, such as graphene oxide. The structure and properties of graphene oxide, a two-dimensional carbon-based material with unique properties and potential applications in a variety of fields, including materials science, nanotechnology, and biotechnology, are better understood thanks to FT-IR analysis, which offers insightful information into the vibrational modes of molecules [18]. In contrast, C=N stretching vibrations and skeletal motions from unoxidized graphitic domains are indicated by the bands at 1706 cm and 1041 cm, respectively (Fig:5.4). O-H stretching vibrations are represented by the strong band at 3294 cm. The band at 1035 cm suggests vibrations caused by C-O stretching. Additional characteristics include the peak at 1043 cm, which could be caused by the -C-O stretching frequency, and the peak at 1571 cm, which corresponds to the vibration spectrum of the carbon-carbon double bond. However, a second prominent peak that was seen at 1704 cm lends credence to the -C=N stretching frequency.

5.1.4 Scanning Electron Microscopy (SEM) with EDX for $\text{Fe}_3\text{O}_4/\text{GO}$

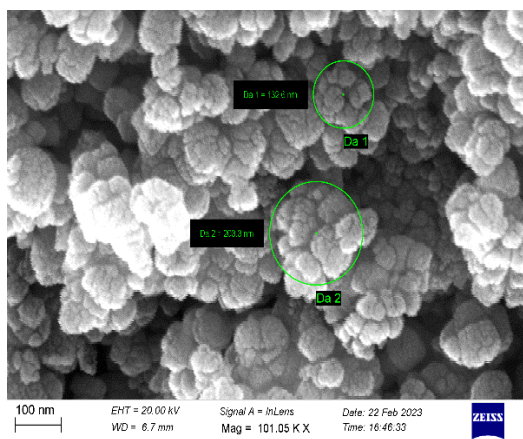


Fig.5.5: SEM of $\text{Fe}_3\text{O}_4/\text{GO}$

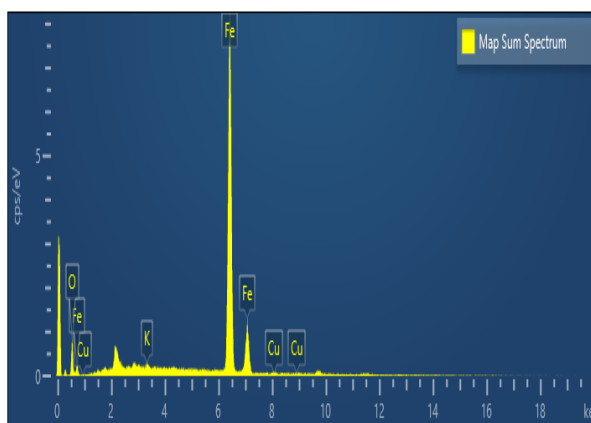
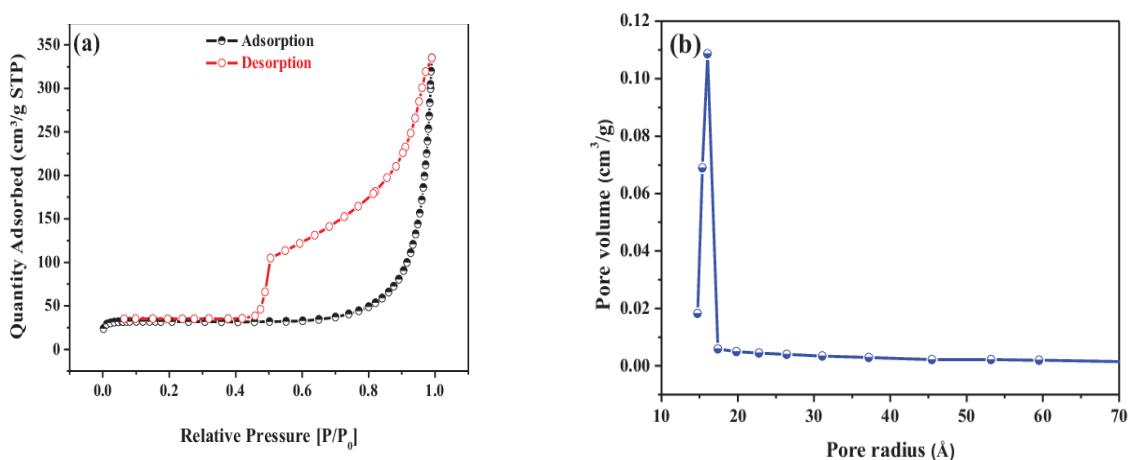


Fig.5.6: EDX of $\text{Fe}_3\text{O}_4/\text{GO}$

For high-resolution imaging and elemental analysis of materials, scanning electron microscopy (SEM) in conjunction with energy-dispersive x-ray spectroscopy (EDX) is a particularly effective analytical method. Numerous domains, including materials science, nanotechnology, geology, forensic science, metallurgy, and others, find major usage for SEM with EDX. A finely focused electron beam analyses the sample surface during SEM with EDX analysis, creating secondary electrons, backscattered electrons, and unique X-rays. In-depth photos of the sample's surface morphology may be observed in the secondary and backscattered electrons, and the unique X-rays reveal essential data about the sample's elemental composition [19]. Scanning electron microscopy was employed to investigate and characterise the synthesised nano-composite. Nanoparticle analysis is done using SEM. (Fig:5.5) was scanned at a distance of 100 nanometres. The scanning electron microscope image makes it obvious that FeSO_4 was successfully attached to the graphene oxide, as evidenced by the porosity network that it generated, which has the appearance of a loose sponge. The nanocomposite's EDX graph is provided in (Fig:5.6).

Table.5.1: EDX composition

Element	Line Type	Weight %	Weight %Sigma	Atomic %
O	K series	17.05	0.20	32.18
K	K series	0.36	0.09	0.46
Fe	K series	67.91	0.34	61.2
C	K series	14.68	0.27	27.64
Total		100.0		100.0

5.1.5. Brunauer-Emmett-Teller analysis**Fig.5.7,5.8: BET isotherm, Pore radius vs volume**

Brunauer-Emmett-Teller (BET) analysis is a well-liked and trustworthy technique for precisely calculating the surface area of materials, such as nanoparticles, porous materials, and catalysts. It is based on the concept of gas adsorption onto the sample surface and provides crucial details on the porosity, pore size distribution, and surface area of materials. BET analysis has applications in many fields, such as materials science, chemistry, chemical engineering, and environmental research, among others, since it provides an accurate estimate of surface area at the nanoscale. The total surface area of the synthesised graphene-based Fe

nanocomposite was estimated using a Brunauer-Emmett-Teller (BET) surface area analyzer and nitrogen adsorption/desorption isotherms at a bath temperature of -196.3°C. The surface area was determined using the BET multi-point and single-point approaches based on the N₂ adsorption/desorption isotherm values. In order to clear the pores before analysis, the graphene-based nickel nanocomposite was vacuum evacuated for a whole night at 150°C. Using the BJH methodology, which integrates the DH method and Kelvin equation, the data on pore size distribution and pore volume were calculated [20]. The BJH model was used to evaluate the pore size distribution, and the Dubinin-Radushkevich (DR) method was used to determine the micropore volumes of the samples. The BET surface area measurement parameters are summarised as follows: 11.0 m²/g of micropores, 5.9 nm pore size, 0.025 cm³/g of pore volume, and 16.1 m²/g of surface area.

5.2 EFFECT OF VARIABLES ON REMOVAL OF Cr⁶⁺

5.2.1 Effect of Nano-Composite Dosage

The effect of adsorbent dosage on the adsorption process is a crucial factor that must be carefully considered. Adsorbent dosage refers to the quantity of adsorbent material used relative to the amount of adsorbate being treated. The optimal dosage of adsorbent has a important impact on the efficiency and effectiveness of this adsorption process [7]. It is essential to accurately determine the appropriate dosage of adsorbent for a specific adsorption system, considering factors such as the characteristics of the adsorbate and adsorbent, desired level of adsorption, and operational parameters. The dosages of 0.5g, 1.0g, 2.0g of weight were taken for consideration. The dosage of 0.5g is best suited for the below shown (Fig:5.9)

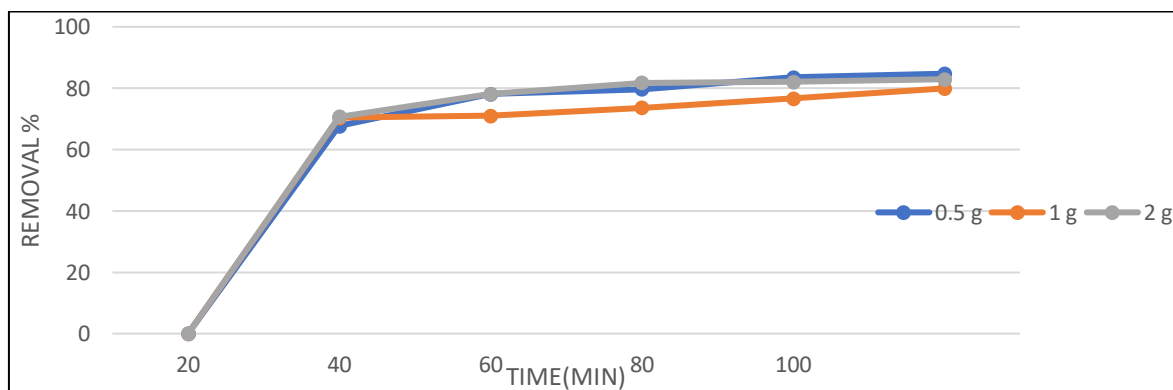


Fig.5.9: Effect of Dosage

5.2.2 Effect of Concentration

A key factor in the adsorption process is the concentration of the adsorbate in the fluid phase. The impact of concentration is concerned with how the adsorbate's concentration affects the adsorbent material's capacity and effectiveness for adsorption. It is important to consider the impact of concentration when designing and optimizing adsorption processes [9]. To find out how the concentration affects the removal percentage of chromium, the range of concentration we use ranges from 100 ppm to 400 ppm, The dosage for the concentration shown (Fig:5.10) is 0.5g.

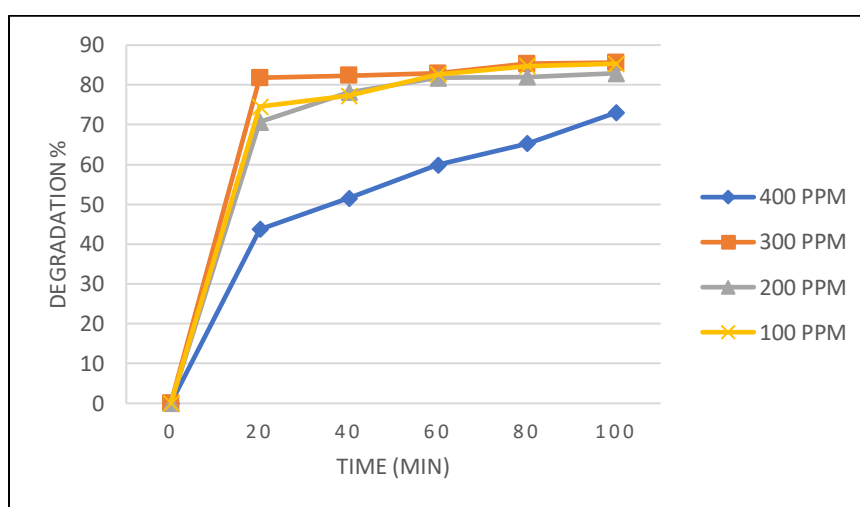


Fig.5.10: Effect of concentration

5.2.3 Effect of Contact Time

The effect of contact time on the adsorption process is crucial, since it impacts the duration that a chemical stay in contact with an adsorbent material. Several parameters, including the type of the adsorbate and adsorbent, temperature, concentration of the adsorbate in the fluid phase, and the desired amount of adsorption, play a critical role in defining the appropriate contact time for adsorption [8]. The impact of contact time at the elimination of Cr^{6+} is indicated in (Fig:5.11). Removal efficiency improves as the duration of the procedure increases. The maximum removal efficiency is found to be 90% at the time of 35 and after that it attains equilibrium.

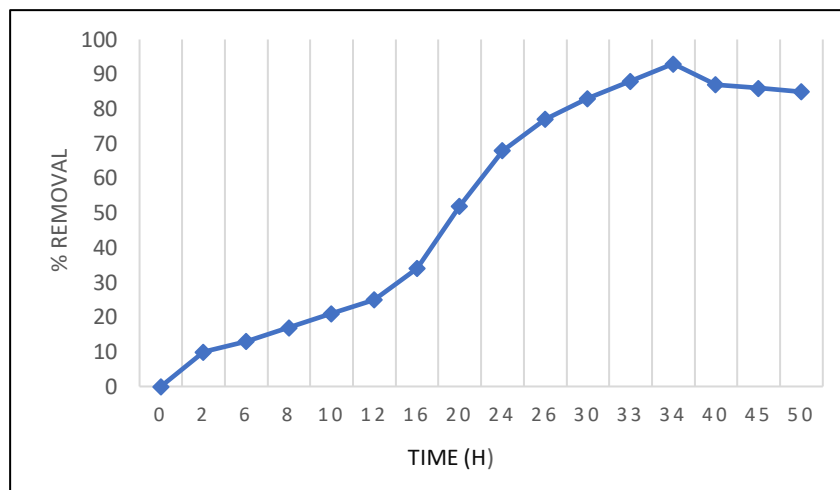


Fig.5.11: Effect Of contact time

5.2.4 Effect of Temperature

The influence of temperature on the adsorption process for chromium removal is important [21]. (Fig:5.12) shows increase in temperature from 40°C to 50°C significantly affect the rate of adsorption owing to the greater kinetic energy of molecules, which promotes enhanced diffusion and mass transfer of chromium ions onto the adsorbent surface. However, if temperature is above 50°C results in decreased adsorption capacity due to changes in surface chemistry, pore structure. The below figure shows the percentage of removal as the temperature varies.

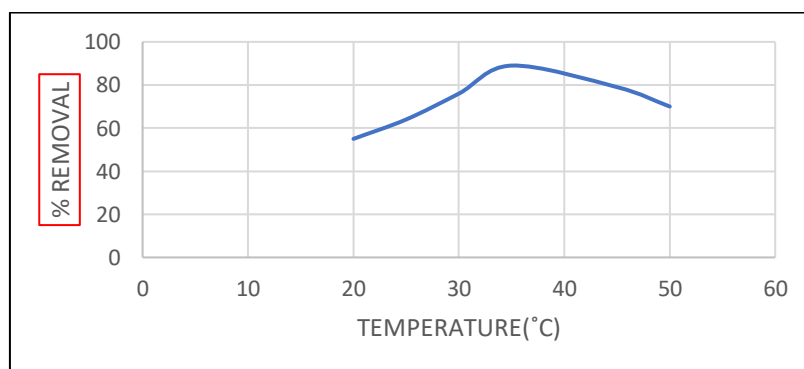


Fig.5.12: Effect of temperature

5.2.5 Effect of pH

The pH of the solutions plays a crucial role in the adsorption process for chromium removal. It disturbs the surface charge of the adsorbent material, the speciation of chromium ions, and the adsorption capacity and efficiency. It is important to consider the pH-dependent effects on these factors when designing chromium removal

processes to ensure originality in the work done [11]. The (Fig:5.13) shown below impacts the pH removal on chromium when undergone through UV radiation. The chromium removal percentage determined with UV light for pH 2, 5, 6.5, 7.5, 10, and 11 were 73, 78, 84, 93, 89, and 77, respectively. So according to the below graph the optimal value of pH is found to be 7.5 as the maximum removal % is found to be 93%.

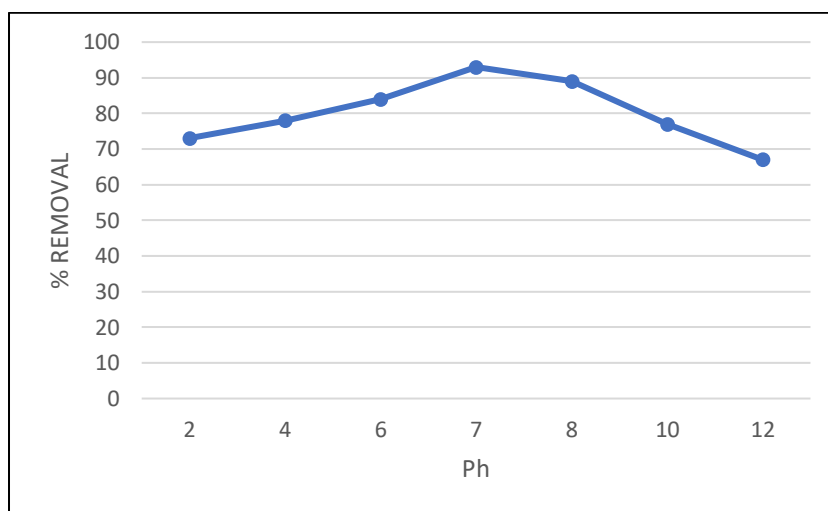


Fig.5.13: Effect of pH

5.3 ADSORPTION ISOTHERM

The distribution of an adsorbate, or the substance being adsorbed, between an adsorbent, or the substance enabling adsorption, and the solution phase at equilibrium, is described by adsorption isotherms, which are mathematical models. These isotherms are often used for modelling and analysis of adsorption processes in a variety of disciplines, including environmental science, chemical engineering, and materials science. They provide insightful information on the behaviour and properties of the adsorbent-adsorbate system [22]. This experimental method makes it easier to determine the maximum adsorption capacity of the adsorbent, which depends on the mathematical model used. The models of Langmuir, Freundlich, and Temkin are some of the ones that are often used to describe solid-liquid sorption systems.

A frequently used model for examining chromium elimination by adsorption is the Langmuir isotherm. It presupposes that there are a limited number of identical adsorption sites on a homogeneous surface in a monolayer. The connection

between the equilibrium adsorption capacity and the equilibrium concentration of the adsorbate in the solution is mathematically described by the Langmuir isotherm. On the other hand, the Freundlich isotherm is a popular adsorption model that is also utilised in the removal of chromium. On a heterogeneous surface, a multilayer adsorption with a variety of adsorption energies is assumed [23]. The mathematical link between the adsorption capacity and the equilibrium concentration of the adsorbate is described by the Freundlich isotherm.

5.3.1 Langmuir Isotherm

A common model in the study of adsorption is the Langmuir isotherm. Irving Langmuir first put out this theory in 1918, and it depicts the adsorption of a solute onto a solid surface as a monolayer with a limited number of adsorption sites [24,25]. The Langmuir isotherm makes the assumptions that all adsorption sites have a constant adsorption energy and that the adsorption process is reversible.

Eqn3 is the Langmuir isotherm equation:

$$q = (q_m K L C) / (1 + K L C) \quad (3)$$

where:

q is the solute adsorbed quantity per unit mass of adsorbent, such as mg/g. The adsorbent's maximal adsorption capacity is q_m (ex.mg/g). Langmuir adsorption constant (L/mg) is KL. c is the solute's equilibrium concentration in the solution (example: mg/L).

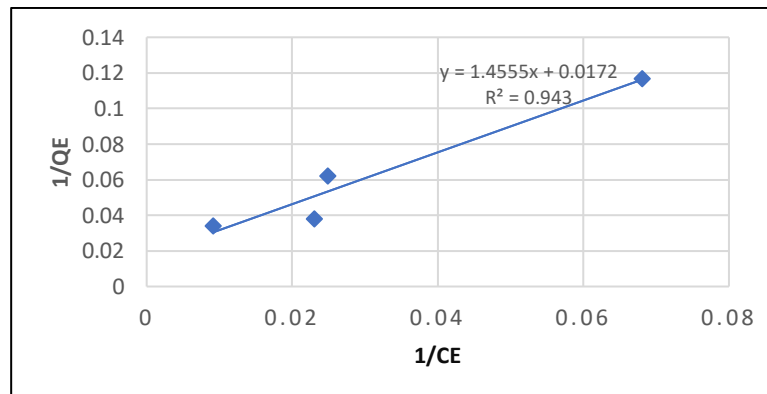


Fig.5.14: Langmuir Isotherm

5.3.2 Freundlich Isotherm

The Freundlich isotherm, proposed by Herbert Freundlich in 1906, is a commonly employed model in this field of adsorption. It describes the adsorption of solutes onto a solid surface with multiple layers of adsorption and assumes a heterogeneous surface with varying adsorption energies for different adsorption sites.

The Freundlich isotherm equation is given by **Eqn4**:

$$q = KFC c ^{(1/n)} \quad (4)$$

where: q is the solute adsorbed quantity per unit mass of adsorbent, such as mg/g. Freundlich adsorption constant (mg/g) / (mg/L)^(1/n) is known as KF. c is the solute's equilibrium concentration in the solution (example: mg/L) [25,27]. The Freundlich exponent (n) characterises the form of the isotherm and symbolises the variability of the adsorption process.

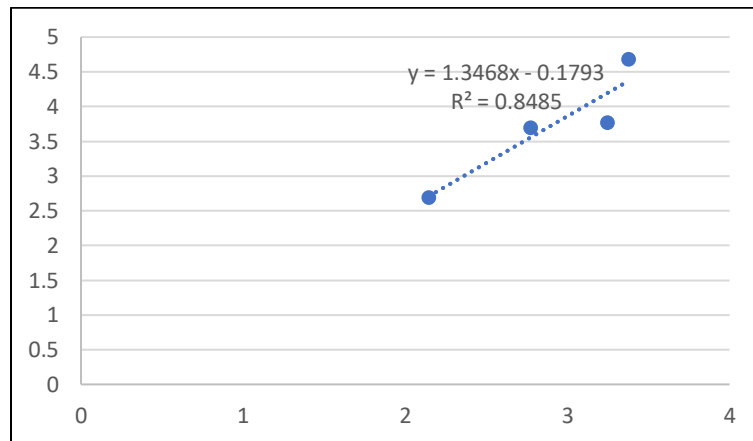


Fig.5.15: Freundlich Isotherm

5.3.3 Tempkin Isotherm

The Tempkin isotherm, originally proposed by M. Tempkin and V. Pyzhev in 1940, is a widely employed model in adsorption studies that accounts for the impact of adsorbate-adsorbent interactions and surface heterogeneity [26]. This model based on the assumption that the heat of the adsorption diminishes linearly with the coverage of adsorbate on the adsorbent surface.

The Tempkin isotherm equation is given by **Eqn 5**:

$$q = B \ln(At ka + 1/B) \quad (5)$$

where:

q is the solute adsorbed quantity per unit mass of adsorbent, such as mg/g. The heat of adsorption (J/mol)-related Tempkin isotherm constant is B . The equilibrium binding constant (L/g) is called At . ka is the adsorption capacity (g/L)-related equilibrium constant.

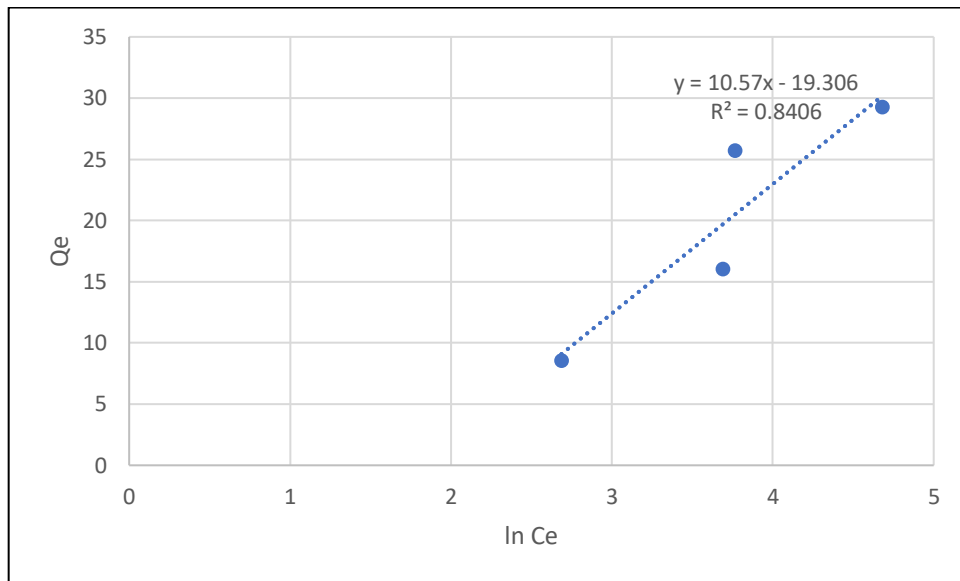


Fig5.16: Tempkin Isotherm

Table5.2: Adsorption Isotherms

Model	Isothermal Constant			
Langmuir	q_e (mg/g)	K_L (L/mg)	R_L	R_2
	42.61	0.015	0.05–0.35	0.943
Freundlich	n	(mg/g)	-	R_2
	2.463	5.69	-	0.848
Tempkin	(L/g)	(mg/L)	(J/mg)	R_2
	0.53	6.78	274.66	0.840

5.4 KINETIC STUDIES OF ADSORPTION

Adsorption kinetic studies are crucial for determining how quickly a material clings to the surface of a solid or liquid, producing a layer. Adsorption plays a crucial role in various scientific and industrial applications, such as catalysis, environmental remediation, and drug delivery. These studies typically involve measuring changes in the concentration or coverage of the adsorbate on the adsorbent over time. This experimental data obtained from these studies are then analyzed to determine the rate of adsorption and the underlying mechanisms, providing insights into the nature of this adsorption process and the factors that affect it [26,27]. By understanding the kinetics of adsorption, we can optimize adsorption processes, design efficient adsorbents, and predict the performance of adsorption systems under different conditions.

5.4.1 Pseudo First & Second Order Reaction

The experimental data for the adsorption of Cr^{6+} were analysed in this work using both the pseudo-first-order and pseudo-second-order models (Fig:5.17, Fig:5.18). The pseudo-first-order and pseudo-second-order figures are provided below. The adsorption of Cr^{6+} was shown to follow a pseudo-second-order kinetic model after comprehensive investigation of the kinetic data. The pseudo-second-order model gave a better match to the experimental data than the pseudo-first-order model, according to a comparison of the kinetic constants derived from the pseudo-first-order and pseudo-second-order graphs. The rate-limiting stage of the adsorption process may entail chemisorption or strong chemical interactions between the adsorbate and adsorbent, according to this suggestion that the adsorption of Cr^{6+} is better represented by a pseudo-second-order kinetic model [28].

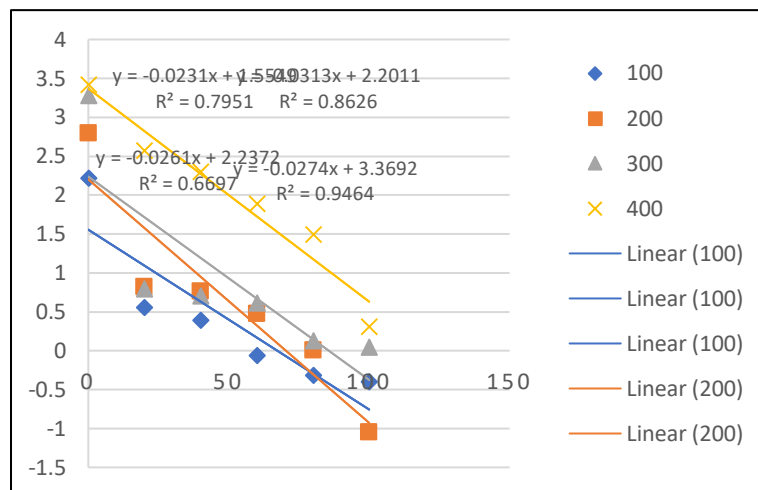


Fig. 5.17: Pseudo First order

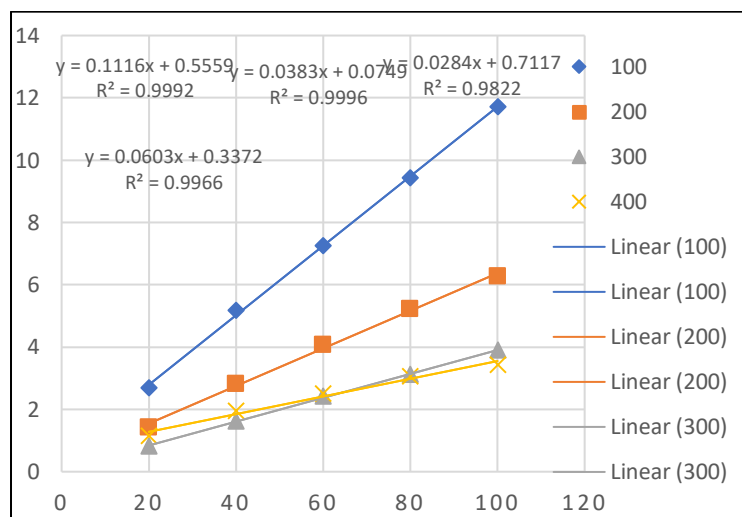


Fig. 5.18: Pseudo second order

Table.5.3: Kinetics of Cr⁶⁺ adsorption

Pseudo 1 st order	k₁ (min⁻¹)	q_e (mg/g)	R²
	0.0214	67.56	0.946
Pseudo 2 nd order	k₂ (min⁻¹)	q_e (mg/g)	R²
	0.00042	58.21	0.996

5.5 MECHANISM OF ADSORPTION

With positively charged chromium ions (Cr) in aqueous solutions, often in the form of Cr^{6+} or Cr^{3+} , graphene oxide (GO), which has a negatively charged surface due to the presence of oxygen functional groups, may exchange ions with them. Since the positively charged Cr ions and the negatively charged surface of GO have a high electrostatic affinity, this results in the substitution of surface oxygen functional groups with Cr ions [29]. Diverse oxygen functional groups, such as hydroxyl (OH), epoxy (C-O-C), and carboxyl (COOH) groups, are abundant on the surface of graphene oxide. These groups promote powerful interactions with chromium ions (Cr) through electrostatic attractions, hydrogen bonds, and van der Waals forces. Therefore, via these interactions, Cr ions may adsorb onto the surface of graphene oxide, leading to the creation of Cr-GO surface complexes [30]. Both graphene oxide (GO) and Fe_3O_4 nanoparticles include oxygen functional groups that may form chemical bonds with chromium (Cr) ions by coordination or chelation, leading to chemical adsorption. The $\text{Fe}_3\text{O}_4/\text{GO}$ composite's surface oxygen functional groups and the Cr ions may form complexes, which effectively removes the Cr ions from the solution.

5.6 COMPARITIVE STUDIES

Table.5.4: Comparison of different adsorbents

Adsorbent	Concentration of adsorbent (g L^{-1})	pH	Contact time (min)	Adsorption capacity (mg g^{-1})	Ref.
Layered zinc hydroxide nitrate	1.5	8	120	194.3	21
Amine-based-surfactant-modified clinoptilite	14	4	120	6.72	14
Superparamagnetic cationic cryogels	0.15	7	180	65.5	20
Magnetic liquid/chitosan/graphene oxide	1.0	3	40	145.35	19

HDTMA-zeolite	25	3	240	8.83	22
Nickel ferrite composite built on graphene oxide	2.5	4	120	44.9	16
Fe₃O₄/GO nanocomposites	1.0	7.5	90	85.6	THIS WORK

5.7 REGENERATION STUDIES

In this work, we showed that the synthesised Fe₃O₄/GO nanocomposite for Cr⁶⁺ adsorption is regenerable and reusable. Initially introduced to a beaker, the adsorbent was given 60 minutes to absorb Cr⁶⁺ while being vigorously stirred. After that, deionized water was used to filter and clean the adsorbent. In order to activate the adsorbent sites and dissolve all adsorbed metal, the utilised adsorbent was heated to around 150°C. The filtrate's absorbance was measured after this cycle was run six times, with each round including 60 minutes of stirring with Cr⁶⁺ metal. The outcomes, as seen in (Fig.5.19), were encouraging. The efficiency of the adsorbent dropped to 87% after the first recycling, which might be due to nanoparticle clustering during sintering. However, the efficiency continued to decline in the same sequence for the second round and following cycles. Despite this, the substance may still be employed for many iterations of adsorption or purification, demonstrating its capacity for regeneration and reusability. In conclusion, our research showed that Fe₃O₄/GO nanocomposite may be recycled and utilised again for Cr⁶⁺ adsorption, however the effectiveness may somewhat deteriorate after many cycles.

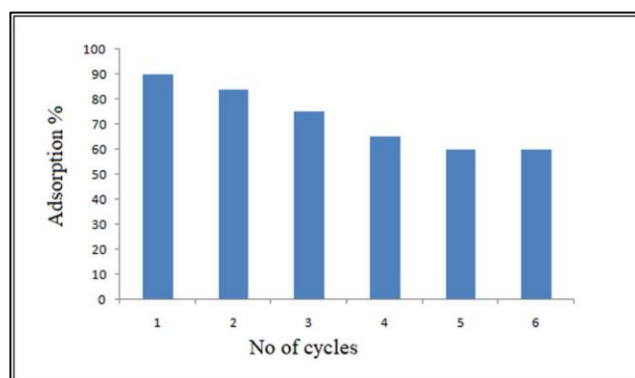


Fig. 5.19: Graph of *Regeneration studies*

CHAPTER-6

SUMMARY AND CONCLUSION

A novel Fe_3O_4 -impregnated graphene oxide $\text{Fe}_3\text{O}_4/\text{GO}$ nanocomposite was successfully prepared using a sustainable methodology and was employed as an efficient adsorbent for removal of Cr^{6+} . The enhanced Removal efficiency of $\text{Fe}_3\text{O}_4/\text{GO}$ was better because of the formation of a heterojunction between Fe_3O_4 and GO, due to its high adsorption capacity and straightforward magnetic separation, $\text{Fe}_3\text{O}_4/\text{GO}$ nano composite has the potential to be an efficient technique to remove heavy metals and other contaminants from wastewater. FE-SEM, XRD, FT-IR and BET were used to characterise the synthesised $\text{Fe}_3\text{O}_4/\text{GO}$ Nano-composite. In order for the heavy metals to adhere to $\text{Fe}_3\text{O}_4/\text{GO}$, coordination bonds between the metal ions and the surface-addressed functional groups must form. Some of the factors considered for this procedure are the adsorbate concentration, pH, temperature, and contact time, by maximising these variables, $\text{Fe}_3\text{O}_4/\text{GO}$'s adsorption capacity might be improved, making it a more effective adsorbent. The highest removal efficiency we achieved by considering all the above mentioned parameters is 85.6%. The isotherm data were analysed by the Langmuir and Freundlich isotherms and the data fitted Freundlich model better than the Langmuir isotherm model. The kinetic data followed the pseudo-second-order kinetic model. The potential $\text{Fe}_3\text{O}_4/\text{GO}$ for reuse is also encouraging. It is regenerable for a maximum of three times, giving degradation percentages of 91%, 87%, and 76%, respectively. The deterioration percentages for the fourth, fifth and sixth are 70%, 68% and 64%, respectively.

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final report 1

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INTRODUCTION

1.1. BACKGROUND AND CONTEXT

A collection of metallic elements with a relatively high density and atomic weight are known as heavy metals. In industrial operations including mining, smelting, and metal refining, they are often utilised. But if released into the environment, they might have a negative impact on both the ecosystem and human health owing to their poisonous nature.

A heavy metal is a metallic element having a relatively large atomic weight, specific gravity, or density. In industrial waste water, heavy metals including lead, cadmium, mercury, cobalt, and others could be present.

It is vital to remove heavy metals from waste water since they do not naturally degrade and are very toxic to aquatic life even in little amounts.

Adsorption is a mass-transfer technique in which the surface of the solid receives the deposition of the dissolved solid from the liquid as a result of physical or chemical contact. When dissolved particles from a liquid attach to the surface of a solid, the process is known as adsorption.

The use of inexpensive adsorbents made from industrial wastes as an alternative to pricey traditional methods of collecting heavy metal ions from wastewater is of great interest to many researchers. Fly ash, waste slurry, red mud, lignin, sugar beetroot pulp, blast furnace slag, tea industry waste and sugar cane bagasse are examples of promising materials.

Utilising two different Turkish fly ashes (Afsin-Elbistan and Seyitomer), nickel [Ni(II)], Cu(II), and zinc [Zn(II)] were removed from an aqueous solution. It was discovered that fly ash from Afsin-Elbistan, which has a high calcium concentration, works just as well as activated carbon as a metal adsorbent. In this study, the effects of four variables—fly ash/lime mass ratio, fly ash/lime absorbent type, solution temperature, and sorbent concentration—were examined in relation to the removal of the five metallic ions (Cu^{2+} , Ni^{2+} , Zn^{2+} , Cd^{2+} , and Pb^{2+}). With a solution temperature of 66 °C and a sorbent ratio of 0.66, they found that an adsorbent with a concentration of 122 g L⁻¹, or fly ash and 5% by mass of lime, produced both high adsorption and low dissolution. Namasivayam tested the effectiveness of

eliminating hazardous anions, dyes, heavy metals, and organic compounds from water using jatropa husk activated carbon generated from agro-industrial solid waste.

According to studies, 97% of nickel and 61% of the original percentage of chromium IV could both be removed. Several studies have been conducted on the removal of heavy metals from waste water by industrial waste. the efficiency of removing certain types of industrial waste.

The investigation into utilising agricultural leftovers as adsorbents to remove heavy metals from industrial wastewater has recently attracted a lot of attention. The method of employing agricultural wastes in the bioremediation of heavy metal ions is known as bio-sorption.

Certain biomass may passively concentrate and quell contaminants onto its cellular structure via to a physiochemical process known as bio sorption. Many scientists believe that this phenomena will provide a practical means of removing dangerous heavy metals from industrial wastewater and will aid in environmental cleaning. Schiewer looked into and compared the ability of pectin-rich fruit peels to absorb cadmium metal and found that grape skin had the highest capacity when compared to orange, lemon, apple, etc. The majority of heavy metals, including Pb^{2+} , Cd^{2+} , Zn^{2+} , Cu^{2+} , and Ni^{2+} , have higher sorption capacities for rice hull than saw dust because of the silica functional group on rice hull and its high surface area, according to a study and comparison of the heavy metal removal capacities of rice hull and saw dust. The biosorption capabilities of raw ruin and ruin resin were 26.3 and 41.6 mg/g, respectively, according to biosorption and desorption studies on chromium(VI) using innovative ruin and ruin resin bio adsorbents. The major functional groups linked to the resin for the aim of eliminating chromium from waste water were the hydroxyl, carboxyl, and carbonyl groups. It was suggested that toxic heavy metal ions could be removed from aqueous solutions using chemically altered agricultural waste (okra biomass). It was discovered that the okra biomass had a very high capacity for adsorption of Pb^{2+} , Cd^{2+} , Cu^{2+} , and Zn^{2+} at respective concentrations of 273.97 mg/g, 121.51 mg/g, and 57.11 mg/g. Seaweed (*Ascophyllum nodosum*), sawdust, and reed plant root (*Phragmites australis*) were three low-cost bio adsorbents that were contrasted. Of the three, sawdust exhibited the most potential for lead adsorption, whereas seaweed showed the best cadmium adsorption capacity. Which heavy metals can be removed through biosorption depends on the functional group attached to the bio adsorbents; for instance, the silica group has a stronger affinity for copper, zinc, and lead than the hydroxyl group does for copper, lead, and nitrogen.

When selecting an adsorbent to treat wastewater, two key features of this technology—technical applicability and economic effectiveness—should be taken into consideration. Adsorbents may be classified as being made of natural materials, man-made materials, agricultural waste, and biological waste.

Materials made of nanoparticles mixed with a polymer matrix or other materials are known as nanocomposites. Large surface area, excellent mechanical strength, and changeable surface chemistry are just a few of their special qualities. These characteristics make them perfect for a variety of uses, including water filtration, medication delivery, and catalysis.

The adsorption procedure is one of the most popular ways to remove heavy metals from water. In this process, heavy metal ions are bound to an adsorbent material's surface. Activated carbon, zeolites, and many kinds of nanoparticles are examples of adsorption materials that may be either natural or artificial.

Nanocomposites have been thoroughly studied as potential adsorbent materials for heavy metal removal. They have a large surface area and a diverse surface chemistry, which may allow them to successfully adsorb heavy metal ions from water. Its properties may also be easily changed in order to improve its capacity to adsorb and select certain heavy metal ions.

One of the most common sources of heavy metal pollution, which is a severe environmental problem, is industrial effluent. Various industrial procedures, such as mining, electroplating, and metal processing, have the potential to discharge significant amounts of heavy metals into the environment. These procedures may also contaminate water sources and harm both human and ecological health.

Adsorption is the method used most often to remove heavy metals from wastewater. Heavy metal ions may be physically or chemically removed from water by the process of adsorption, which involves binding the metal ions to the surface of an adsorbent material. Traditional adsorbents such as activated carbon and zeolites have been used for this purpose, but they have limitations including poor selectivity, low adsorption capacity, and high cost.

Nano-adsorbents composed of nanoscale particles or fibres were investigated as an alternative to conventional adsorbents for the removal of heavy metals from wastewater. These materials have many advantages over traditional adsorbents, including a wide surface area, changeable surface chemistry, and strong selectivity to certain heavy metal ions. Nano

adsorbents may be made from a wide range of materials, such as metal oxides, polymers, and carbon nanotubes.

Recent years have seen a lot of study on ⁶⁰the use of nano adsorbents to remove heavy metals from wastewater. According to studies, these materials may efficiently and selectively remove heavy metals from wastewater, including lead, cadmium, and mercury. Furthermore, compared to conventional adsorbents, the usage of nano adsorbents may lead to reduced prices and quicker removal times. However, using nano adsorbents to remove heavy metals from wastewater comes with a number of difficulties. The potential toxicity of these compounds is one of the main obstacles. According to certain research, releasing certain kinds of nanosorbents into the environment might be harmful to human health. It is crucial to guarantee the appropriate handling and disposal of these items.

The expansion of laboratory technologies to an industrial scale presents another difficulty. Nano adsorbent synthesis and manufacture may be difficult, requiring specialised knowledge and tools. Therefore, for the widespread use of nano adsorbents in industrial applications, the development of affordable and scalable manufacturing procedures is crucial.

Despite these challenges, there are some advantages to employing nanoadsorbents rather than traditional adsorbents to remove heavy metals from wastewater. They offer high selectivity, efficiency, and adsorption capacity and are easily tailored to specific applications. Since they may also shorten the cost and time of the heavy metal removal procedure, they are a promising option for treating industrial wastewater.

Using adsorption methods to remove heavy metals from nanocomposites is a key area of research with the aim of developing efficient and economical methods for removing heavy metals from water. This area of research has a big impact on the environment and people's health, and it has the potential to enhance the development of environmentally friendly and sustainable technologies.

In conclusion, the research of employing nano adsorbents to remove heavy metals from wastewater is an area that is rapidly increasing and has tremendous potential to have a good impact on both the environment and the economy. Although there are drawbacks to this approach, further investigation and development may lead to the widespread use of nano adsorbents as a cost- and sustainably-effective method of reducing heavy metal pollution.

During the adsorption procedure, pollutants are removed from a solution by binding contaminants to the surface of Fe₃O₄-GO nanoparticles. Fe₃O₄-GO has unique qualities, such as a large surface area, magnetic capabilities, and chemical stability, which make it a viable adsorbent for a variety of applications.

The adsorption process in Fe₃O₄-GO is predominantly driven by the physical and chemical interactions between the molecules of the adsorbate and the adsorbent surface. Examples of physical interactions include van der Waals forces, hydrogen bonds, and electrostatic forces, while chemical interactions include covalent or ion-dipole bonds.

The ability of Fe₃O₄-GO to adsorb is influenced by a variety of variables, including the concentration of the adsorbate, pH, temperature, and contact time. Fe₃O₄-GO's adsorption capacity may be increased by optimising these variables as well as by functionalizing or doping the adsorbent's surface.

Fe₃O₄-GO has undergone considerable research for the removal of numerous impurities from water, including heavy metals, organic pollutants, and pigments. The adsorption of heavy metals onto Fe₃O₄-GO requires the establishment of coordination bonds between the metal ions and the functional groups on the surface of the adsorbent. Fe₃O₄-GO has a great potential for adsorbing heavy metals including lead, cadmium, and mercury, as has been shown.

Adsorption of organic pollutants onto Fe₃O₄-GO includes physical adsorption of the contaminants onto the surface of the adsorbent. The ability of Fe₃O₄-GO to effectively adsorb a number of organic pollutants, including as dyes, phenols, and polycyclic aromatic hydrocarbons (PAHs), has been shown.

In conclusion, Fe₃O₄-GO is an appropriate adsorbent for the removal of pollutants from water because of its enormous surface area, magnetic characteristics, and chemical stability. Fe₃O₄-GO's characteristics and synthesis must be enhanced for certain applications, and further study is required to see if it can remove other pollutants from water.

Fe₃O₄/GO nanocomposites are a kind of hybrid material that has drawn a lot of attention in the field of nanotechnology due to its peculiar blend of magnetic and electrical properties. While graphene oxide alone has high mechanical and electrical conductivity, Fe₃O₄ nanoparticles in the matrix provide the material stronger magnetic properties. Graphene oxide is commonly reduced in the presence of Fe₃O₄ nanoparticles to produce Fe₃O₄/GO

nanocomposites. The finished item has unique ³ properties that make it desirable for a variety of applications, including biomedical imaging, medicine delivery, and environmental remediation.

Biomedical imaging is one of Fe₃O₄ nanocomposites' most important uses. The graphene oxide matrix may be functionalized with targeted molecules to increase imaging specificity, and Fe₃O₄ nanoparticles can be used as contrast agents for MRI due to their magnetic characteristics. Additionally, Fe₃O₄/GO nanocomposites have significant promise as drug delivery methods. Utilising magnetic fields, Fe₃O₄ nanoparticles may be utilised to direct nanocomposites to certain body locations, while functionalizing the graphene oxide matrix can improve drug loading and release.

Fe₃O₄/GO nanocomposites have also been investigated for their potential in environmental remediation. While the graphene oxide matrix may be utilised to absorb pollutants and contaminants, the magnetic characteristics of Fe₃O₄/GO nanoparticles can be employed to separate nanocomposites from contaminated water.

Energy storage is another another possible use for graphene oxide-Fe₃O₄ nanocomposites. Graphene oxide has a high electrical conductivity and a big surface area, making it a great material for supercapacitors. The nanocomposite material created by adding Fe₃O₄ to the graphene oxide matrix exhibits enhanced capacity and cycle stability, making it a strong contender for high-performance energy storage devices.

Additionally, Fe₃O₄ has been researched for its potential in catalysis. The graphene oxide matrix serves as a stable and conductive substrate for the Fe₃O₄ particles, which may function as effective catalysts for a variety of chemical processes.

Advances in nanotechnology and materials science have made it easier to create Fe₃O₄/GO nanocomposites. The production of nanocomposites with customised characteristics for particular purposes has been made possible by the ability ³ to regulate the size, shape, and distribution of the nanoparticles inside the graphene oxide matrix. Fe₃O₄/GO nanocomposites, in general, provide a special mix of characteristics that appeal to a wide range of applications. Although more study is required to completely comprehend their potential qualities and uses, they have the potential to enhance our everyday lives in a number of ways. Bio adsorbents, low-cost adsorbents, ¹ and new adsorbents have been the most frequently studied adsorbents in recent years ¹ for the treatment of wastewater contaminated with heavy metals; however, other than modified natural adsorbents, no adsorbents have been

used industrially to remove heavy metals. The approach is not in any way scaled up or made available to the industry. The study's only goal has been to determine whether or not that substance is suitable for getting rid of heavy metals and, if so, how successful it is at doing so. There hasn't been a thorough examination of the desorption process and disposal of the adsorbent utilised to extract heavy metals. Since many of them have stated, although pre-treatment and expensive handling are still required for the disposal of sludge, adsorbents like industrial waste, agricultural waste, and biological waste all have excellent adsorption capacity for the removal of heavy metals and are also readily available.

Further study is required to lower the cost of pre-treatment, regeneration, and disposal. The primary field of research for the removal of heavy metals is without a doubt the utilisation of readily available adsorbents.

1.2. STATEMENT OF THE PROBLEM AND RESEARCH QUESTION

Heavy metals in industrial waste water pose a serious threat to the environment, and their removal is crucial to avoid pollution and safeguard public health. Precipitation, ion exchange, and filtration through membranes are examples of traditional treatment techniques, although they have drawbacks such high cost, poor efficiency, and limited scalability. Therefore, a low-cost and efficient method of extracting heavy metals from industrial effluents is required.

Adsorption is a potential method for removing heavy metals because of its great effectiveness, cheap cost, and simplicity of use. Due to their substantial surface area and strong magnetic characteristics, Fe₃O₄/GO nanocomposites have shown remarkable heavy metal adsorption abilities. However, the effectiveness of these nanocomposites for the removal of heavy metals from industrial wastewater has not yet been completely investigated, and in order to maximise efficiency and scalability, it is necessary to optimise their synthesis and working conditions.

The goal of this work is to evaluate the efficacy of Fe₃O₄/GO nanocomposites for the adsorption-based removal of heavy metals from industrial effluents as well as to maximise the synthesis and operating conditions for scalability and maximum efficiency.

This work covers other significant aspects impacting heavy metal adsorption by graphene oxide-Fe₃O₄ nanocomposites in addition to optimising the production and operation conditions. The pH of the solution, the initial concentration of heavy metals, the amount of

time **the** nanocomposites have to come into contact with the effluent, and the existence of other competing ions are some of these variables.

A series of tests will be performed as part of the research to assess the effectiveness and adsorption capacity of graphene oxide-Fe₃O₄ nanocomposites for different heavy metals, including chromium(VI). To identify the ideal circumstances for maximal adsorption, the effect of several factors, including pH and starting concentration, is researched.

Graphene oxide-Fe₃O₄ nanocomposites may be used **52** **to remove heavy metals from** industrial effluent, according **to the** findings **of** this research. The creation of an affordable and effective adsorption method that can be used on a wide scale is made possible by the optimised chemistry and operating conditions. As a result, the issue of heavy metal pollution in industrial effluents will be addressed and a greener, more sustainable approach to wastewater treatment will be developed.

- What are the Fe₃O₄/GO nanocomposites' chromium adsorption capability and efficiency in aqueous solutions?
- How do the solution pH, starting heavy metal content, and contact duration affect the effectiveness of Fe₃O₄/GO nanocomposites' ability to adsorb substances?

Is it possible to regenerate and reuse Fe₃O₄/GO nanocomposites for numerous adsorption cycles?

- What impact do competing ions in industrial wastewater have on Fe₃O₄/GO nanocomposites' adsorption efficiency? What are the Fe₃O₄/GO nanocomposites' chromium adsorption capability and efficiency in aqueous solutions?
- How do the solution pH, starting heavy metal content, and contact duration affect the effectiveness of Fe₃O₄/GO nanocomposites' ability to adsorb substances?

Is it possible to regenerate and reuse Fe₃O₄/GO nanocomposites for numerous adsorption cycles?

- What impact do competing ions in industrial wastewater have on Fe₃O₄/GO nanocomposites' adsorption efficiency?

Insights into the potential of Fe₃O₄/GO **5** **nanocomposites for the removal of heavy metals from industrial effluents** will be gained from the answers to these research questions, which will also aid in the optimisation of synthesis and operating conditions for optimal

effectiveness and scalability. The research will also assist to the creation of an improved sustainable and environmentally friendly approach to wastewater management by offering useful information on the practical use of nanocomposites in industrial wastewater treatment.

1.3. OBJECTIVES OF THE STUDY

This study's objective is to determine how well a nanocomposite adsorbent removes heavy metals from wastewater with an emphasis on the material's capacity, selectivity, and reusability. To establish if the nanocomposite has the potential to be a workable solution ²⁵ for the removal of heavy metal from wastewater, the objective is to optimise the process parameters and assess the adsorption kinetics and equilibrium isotherms.

The study's other goal is to pinpoint the critical variables, such as pH, contact duration, temperature, starting heavy metal concentration, and adsorbent dose, that impact the nanocomposite's ability to adsorb. The effectiveness of the nanocomposite is evaluated in comparison to other adsorbents often ³⁴ used for the removal of heavy metals from wastewater.

Additionally, ³ the research will use analytical methods including X-ray diffraction (XRD), Fourier-transformed infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) to look into the adsorption process of heavy metals on the nanocomposite surface. The manufacture and characterization: To make GO, one must first synthesise it. Then, using a variety of ¹ analytical methods, such as X-ray diffraction, Fourier transform infrared spectroscopy, transmission electron microscopy, atomic force microscopy, and Raman spectroscopy, one must characterise GO's physicochemical characteristics, including its structure, size, morphology, and surface chemistry.

Functionalization is the process of altering the surface of GO by functionalizing it with different chemical groups or biomolecules in order to improve its properties or tailor its surface for specific applications. Covalent or non-covalent functionalization may be involved, and the study might examine how functionalization affects GO properties.

Uses: Investigate the potential applications of GO ¹¹ in a variety of fields, including as catalysis, energy storage, sensing, biological applications, and environmental cleanup. The study may

include analysing how GO-based materials function in certain applications and determining how effective and efficient they are.

Analysis of the toxicity and environmental effects of GO and its derivatives in a variety of contexts, including in vivo and in vitro research as well as environmental fate and transport investigations. This kind of study aims to identify potential problems associated with the use and disposal of GO and to provide solutions to mitigate such risks.

The outcomes of this research are anticipated to provide useful knowledge on the usage of nanocomposites as an efficient and long-lasting technique for heavy metal removal from wastewater. The findings of this research may be utilised to build an efficient and affordable heavy metals treatment procedure for industrial wastewater.

The research will also assess if it is feasible to regenerate and reuse the nanocomposite adsorbent, which may lower the cost of the treatment procedure overall and lessen its environmental effect.

This study's overarching goal is to create an effective and sustainable process for removing chromium (VI) from wastewater using a nanocomposite adsorbent. This may assist preserve the public's health and address rising concerns about environmental heavy metal contamination. The findings of this research may be utilised to enhance the development of environmentally friendly wastewater treatment systems as well as to encourage the use of nanotechnology in environmental applications.

1.4. SIGNIFICANCE AND POTENTIAL CONTRIBUTIONS OF THE STUDY

The potential contributions that this work might bring to the fields of wastewater treatment and environmental protection are what give it its significance. ¹² Due to their toxicity and endurance in the environment, heavy metals are a major environmental issue, and their presence in wastewater may have negative effects on ecosystem and human health.

Therefore, it is crucial to create sustainable and efficient removal techniques.

³ This work may help shed light on the efficiency and viability of the promising strategy of using nanocomposite adsorbents to extract heavy metals from wastewater. The research might aid in the creation of a heavier metal-contaminated wastewater treatment method that is more successful and affordable, especially in industrial settings.

Engineers, scientists, and decision-makers may utilise ⁵⁶ the findings of this study to optimise the use of nanocomposite adsorbents in wastewater treatment and encourage the use of ecologically benign, long-lasting heavy metal removal methods. The research may also help with the creation of rules and guidelines to deal with heavy metal contamination in the environment, which will eventually preserve the health of people and ecosystems.

Overall, the results of this research might have a big impact on environmental engineering and help create a more sustainable and healthy environment for everyone.

The research may also help to advance the use of nanotechnology in environmental applications. Large surface area, strong reactivity, and excellent selectivity are just a few of the distinctive characteristics that nanocomposite adsorbents have that make them very efficient in adsorbing heavy metals from wastewater. This work may provide important ¹⁹ insights into the usage of nanotechnology in environmental applications by examining the mechanism of heavy metal adsorption on the nanocomposite surface.

The research may also aid in the creation of a circular economy. The total cost of the treatment procedure may be decreased, and the environmental effect of the adsorption process can be reduced, by regeneration and reuse of the nanocomposite adsorbent. This may support the idea of a circular economy, which minimises waste and uses waste materials as resources.

Finally, the course may help future generations of engineers and scientists gain knowledge and skills in the area of environmental technology. The findings of this study may be utilised to create fresh, creative methods for removing heavy metals from wastewater, which may inspire more study and invention in this area.

In conclusion, this work has a wide range of importance and possible contributions. The research may help the development of sustainable and green technologies and contribute to a healthier and more sustainable environment by providing useful information on the usage of nanocomposite adsorbents to remove heavy metals from wastewater.

CHAPTER.2

LITERATURE SURVEY:

A severe environmental issue, heavy metal contamination may impair both the ⁸ecology and people's health. As a result, several techniques, such as adsorption procedures, have been developed to remove heavy metals from wastewater. Nano-composites have been a popular adsorbent for heavy metal removal in recent years because of its large surface area, cheap cost, and customizable characteristics.

Numerous research have looked ⁵ the usage of nano-composites for adsorption-based heavy metal removal from wastewater. For the removal of lead ions from wastewater, Chakraborty et al. (2020) created a chitosan/multi-walled carbon nanotube composite. The findings demonstrated that the composite could remove up to 98% of lead ions from wastewater and had a high adsorption capability.

Ates et al. (2019) created a nano-composite ⁸ for the removal of cadmium ions from wastewater based on graphene oxide and magnetic nanoparticles in a different research. The ⁵⁷ results showed that the composite could remove up to 95% of cadmium ions from wastewater and had a high adsorption capability.

In addition, Yadav et al. (2020) created a composite ²⁵ for the removal of nickel ions from wastewater that ^{is} based on activated carbon and zinc oxide nanoparticles. According to the findings, the composite exhibited a strong capacity for adsorption and could filter up to 96% of nickel ions from wastewater.

Additionally, other studies have looked at the usage of different nano-composites for wastewater heavy metal removal. For instance, Hasan et al. (2020) utilised a composite based on activated carbon and titanium dioxide to remove chromium ions from wastewater, while Njoku et al. (2020) used a composite based on chitosan and activated carbon to remove copper ions from wastewater.

⁵ For the removal of arsenic from wastewater, Wang et al. (2020) created ¹¹ new adsorbent made of magnetic iron oxide nanoparticles and altered graphene oxide. The results demonstrated that the adsorbent had a high capacity for adsorption and could eliminate up to 99.9% of arsenic from wastewater.

To remove cadmium (Cd) from wastewater, Zhang et al. (2020) created a nanocomposite of manganese oxide and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cd ions, and that the pH and starting Cd concentration had an impact on the removal effectiveness.

A composite material based on graphene oxide and manganese dioxide was created in a research by Li et al. (2019) to remove lead ions from wastewater. According to the findings, the composite could remove up to 95% of lead ions from wastewater and had a high adsorption capability.

To remove lead (Pb) from wastewater, Wu et al. (2019) created a nanocomposite ³ of reduced graphene oxide and iron oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Pb ions, and that the pH, contact duration, and starting Pb concentration had an impact on the removal efficiency.

The removal of cadmium (Cd) from wastewater was examined by Chen et al. (2021) using a nanocomposite of chitosan and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cd ions, and that the pH, contact duration, and starting Cd concentration had an impact on the removal efficiency.

To remove lead (Pb) from wastewater, Keshavarz et al. (2020) created a nanocomposite of iron oxide and activated carbon. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Pb ions, and that the pH and starting Pb concentration had an impact on the removal efficiency.

In order to remove nickel (Ni) from wastewater, Kumar et al. (2018) created a manganese dioxide and graphene oxide nanocomposite. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Ni ions, and that the pH, contact duration, and starting Ni concentration had an impact on the removal efficiency.

To remove chromium (Cr) from wastewater, Deng et al. (2017) created a nanocomposite using titanium dioxide and activated carbon. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cr ions, and that the pH, contact duration, and starting Cr concentration had an impact on the removal efficiency.

Aluminium oxide and carbon nanotubes were combined to create a nanocomposite by Gao et al. (2017) for the removal of arsenic (As) from wastewater. The findings demonstrated that the nanocomposite had a high capacity for adsorbing As ions, and that the pH, contact duration, and starting As concentration had an impact on the removal efficiency.

Magnetic carbon nanotubes and chitosan were combined to create a nanocomposite that Li et al. (2021) used to remove mercury (Hg) from wastewater. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Hg ions, and that the pH and starting Hg content had an impact on the removal efficiency.

To remove cadmium (Cd) from wastewater, Shao et al. (2020) created a nanocomposite of zinc oxide and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cd ions, and that the pH and starting Cd concentration had an impact on the removal effectiveness.

Similar to this, Wang et al. (2021) created a composite for the removal of copper ions from wastewater that is based on magnetic graphene oxide and Fe₃O₄ nanoparticles. The findings demonstrated that the composite could remove up to 98% of copper ions from wastewater and had a high adsorption capability.

Using iron oxide and montmorillonite, Wang et al. (2018) created a nanocomposite for the removal of copper (Cu) from wastewater. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Cu ions, and that the pH and starting Cu concentration had an impact on the removal efficiency.

To remove lead (Pb) from wastewater, Nair et al. (2017) created a nanocomposite of polyaniline and graphene oxide. The findings demonstrated that the nanocomposite had a high capacity for adsorbing Pb ions, and that the pH and starting Pb concentration had an impact on the removal efficiency.

Additionally, Chen et al. (2018) developed a composite material based on graphene oxide and hydroxyapatite to remove lead ions from wastewater. The results demonstrated that the composite had a strong adsorption capacity and could remove up to 97% of lead ions from wastewater.

Overall, using nano-composites for adsorption methods ³³to remove heavy metals from ³³wastewater shows encouraging results. To effectively remove heavy metals from wastewater, additional study ^{is} necessary to optimise the synthesis and characteristics of nano-composites.

CHAPTER.3

AIM:

This work intends to ³test the performance of a newly synthesised nanocomposite material for the adsorption-based removal of heavy ¹⁰metals from wastewater. In order to discover whether ^{the} nanocomposite material is effective ^{in removing heavy metals from wastewater}, batch adsorption ^{studies} will be utilised to assess the material's physical and chemical features. The study also attempts to compare the effectiveness of the nanocomposite material with other conventional adsorbents for heavy metal removal and to improve the adsorption process by adjusting parameters like pH, contact length, and adsorbent dosage.

The study will also examine the reusability and stability of the ^{nanocomposite} material for repeated adsorption cycles, create a theoretical model to explain ^{the adsorption process and} establish ^{the} kinetic and ^{thermodynamic parameters}, and show that ^{the} nanocomposite material can actually be ^{used to remove heavy metals from real} wastewater ^{samples}. ^{The} research will also shed light on the process through which heavy metals adhere to the nanocomposite material.

SCOPE:

³The ^{goal of this research} project ^{is to examine the} use ^{of} a special nanocomposite ^{material for} ^{the} adsorption-based removal of heavy metals from wastewater. ^{The} research will concentrate on determining the ideal circumstances for using the nanocomposite material in wastewater

heavy metal removal, how different factors ⁵ affect the efficiency of the adsorption process, and how well the nanocomposite material removes various kinds ⁵ of heavy metals from wastewater. The material's stability and reusability will be evaluated via a number of adsorption cycles.

A variety of analytical approaches will be used in surface characterisation tests to further our knowledge of how heavy metals interact with the nanocomposite material. The findings of this work will have practical consequences for developing solutions for treating wastewater that contains heavy metals, a significant problem in many businesses, that are both ecologically sound and economically viable. However, only laboratory-scale research will really use the nanocomposite material in large-scale industrial wastewater treatment systems.

⁶

CHAPTER.4

4. MATERIALS AND METHODS

4.1. Materials:

The chemical engineering department of Sathyabama University in Chennai provided the ³⁷ hydrogen peroxide (H_2O_2), sodium nitrate ($NaNO_3$), potassium permanganate ($KMnO_4$), sulphuric acid (H_2SO_4), expandable graphite powder (acid washed), $FeCl_2 \cdot 4H_2O$, $FeCl_3 \cdot 6H_2O$, NaOH, and hydrochloric acid (HCl). The pH of the utilised solution was altered by adluting H_2SO_4 or NaOH solutions in the required amounts. The ²⁴ concentration of the solutions was determined using a UV-visible spectrophotometer. For this experiment, only materials of the highest quality were used. ²⁶ Deionized water was used throughout the experiment.

4.2. Graphene-Oxide Synthesis

Graphite flakes were employed as the starting material in a modified Hummers method to create GO nanoparticles. The right synthesis techniques and level of oxidation determine the structure and characteristics of GO. 2g of sodium nitrate and 2g of graphite flakes were mixed for 4 hours in 90 mL of H_2SO_4 (fig. 4.1). This solution was gradually given 12 g of $KMnO_4$ while the temperature was maintained below $10\text{ }^{\circ}C$. After another hour of stirring, the resultant liquid was progressively diluted. (184 mL). The next two hours were spent stirring this mixture at $35\text{ to }40\text{ }^{\circ}C$ until it became pasty brown. After mixing, the mixture was held at $25\text{ }^{\circ}C$ for two hours. By adding 200 mL of fresh water and 48% of the H_2O_2 solution (40 mL), the reaction was stopped. Using deionized water and a 10% HCl aqueous solution, the mixture was rinsed until the pH of the final solution was neutral. The mixture was then sonicated after that. To create GO nanoparticles, the precipitate is dried for 24 hours at $60\text{ }^{\circ}C$.



4.3. GO/ Fe_3O_4 Magnetic Nanocomposite Synthesis

In a one-pot simplified co-precipitation technique, superparamagnetic Fe_3O_4 nanoparticle production and GO reduction were carried out simultaneously to create RGO/ Fe_3O_4 magnetic nanocomposites. First, 150 mL of distilled water was combined with 50 mg of GO and put through a two-hour ultrasonic dispersion. Second, 50 mL of deionized water was used to dissolve 176 mg of $FeCl_3 \cdot 6H_2O$ and 130 mg of $FeCl_2 \cdot 4H_2O$ before being added to the GO solution. Once the pH of the solution reached 10, the solution containing sodium hydroxide was introduced drop by drop. The mixture was then swirled for two hours while being heated to $80\text{ }^{\circ}C$. After the mixture had been evenly distributed, the solution of 300 mg of $NaBH_4$ in 50 mL of purified water was gradually added while the mixture was being agitated. At $80\text{ }^{\circ}C$, this treatment was then continued for an extra hour. After being magnetically separated, the

finished product (RGO/Fe₃O₄ nanocomposite) underwent many cleaning steps using distilled water and ethanol before being dried at 50 degrees Celsius.



4.4. Preparation of dye solution for batch study

100 mg/L stock solutions of the chromium (VI) metal were produced. Additionally, working standard solutions were created every day from the stock solutions by appropriate dilution with deionized water. Adsorption tests were routinely carried out in batch manner. A magnetic stirrer was used to mix the adsorbent and 100 millilitres of the stock solution in a 500 ml beaker. At room temperature, the stirring rate was held constant at 250 rpm and the working temperature was set at 25 C. The pH of the solution was changed by adding HCl or an alkali solution, and the new pH was then measured using a pH metre. Using filter paper, the adsorbent nanoparticles were removed from the solution. The absorbance of the test sample was monitored over several time periods to look for variations. The metal concentration in this study was determined using an ultraviolet-visible (UV-Vis) spectrophotometer. You can determine the adsorption capacity by using the Eqn (1,2)

$$q_e = [(C_0 - C_e)/V] M \quad (1)$$

$$\% \text{ Adsorption} = (C_i - C_e)/C_i \quad (2)$$

q_e amount of metal adsorbed in mg per gram of adsorbent.

C_0 and C_e (mg/L) are initial and equilibrium metal concentrations.

²
 C_i and C_e are initial and equilibrium concentration of Cr (VI) (mg/L), respectively.

V is the volume of solution.

M is the mass of adsorbent.

4.5. XRD, FTIR, TEM and SEM study

³
The Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Transmission Electron Microscopy (TEM), and Brunauer-Emmett-Teller analysis (BET) were only a few of the analytical techniques used.

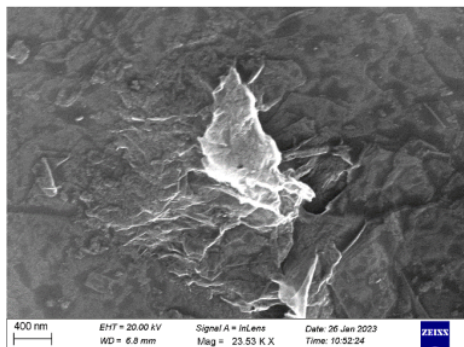
4.6. Isotherms, Kinetics and Thermodynamic Study

⁷
The data from Cr (VI) adsorption equilibrium were fitted using the Freundlich, Langmuir, and Temkin model. The analysis of equilibrium data required fitting the data into linear equations and determining the constants in various isotherm equations. We used linear forms of pseudo-first-order, pseudo-second-order, and intraparticle diffusions to estimate the rate of Cr (VI) adsorption onto Fe₃O₄/GO. Entropy, enthalpy, and free energy calculations were used to study the kinetics of Cr (VI) adsorption.

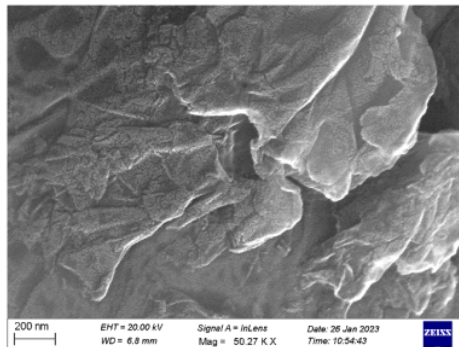
¹³ 5. Results and discussion

5.1 Characterization of graphene oxide

5.1.1. Scanning electron microscopy (SEM)



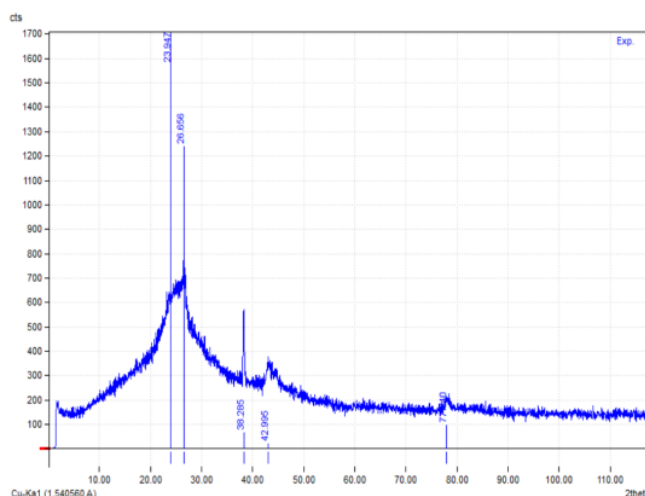
(fig.5.1)



(fig.5.2)

Scanning electron microscopy (SEM) analysis is a powerful imaging and analytical technique that employs a focused electron beam to examine the surface morphology, elemental composition, and other properties of a sample with exceptional resolution. SEM analysis has wide-ranging applications in various fields, including materials science, nanotechnology, biology, geology, and forensic science, among others, owing to its versatility and precision in characterizing different types of samples. Graphene oxide's scanning electron micrograph (Fig. 5.1) reveals an extremely uneven and heterogeneous surface structure. However, graphene has a layer structure at its core. When fictitiously modelled, GO displays a three-dimensional network of randomly aligned sheet-like structures with a stretched texture and centralized pores with a wide size distribution, which may be the best locations for heavy metal binding, forming a porous structure with a loose sponge-like shape that indicates the oxidation process at graphene has occurred. The (fig.5.2) shows in the below is a scanned image at 200 nm.

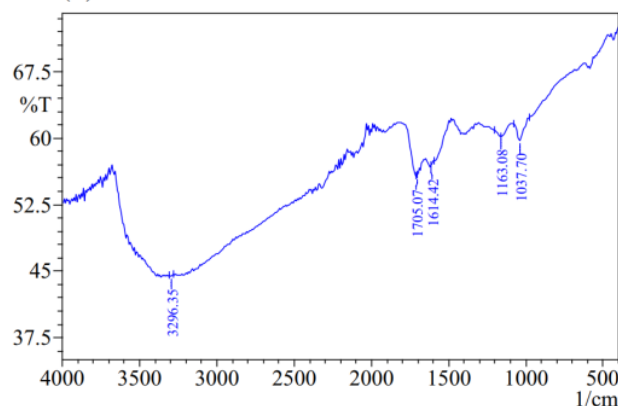
5.1.2. X-ray diffraction (XRD) study



(fig.5.3)

55 X-ray diffraction (XRD) analysis is a non-destructive and highly informative technique that employs X-ray beams to investigate the crystal structure, phase composition, and other properties of materials. Due to its ability to provide valuable insights into the atomic and molecular arrangement of materials, XRD analysis finds widespread applications in various fields, including materials science, solid-state physics, chemistry, geology, and pharmaceuticals. When applied to graphene oxide, XRD analysis can yield crucial information about the interlayer spacing, crystallite size, and stacking arrangement of graphene oxide layers, which are key to understanding its structural properties. The interlayer spacing can be determined from the position of the diffraction peak, providing insights into the degree of oxidation and the presence of the functional groups on the graphene oxide layers. Graphene oxide's XRD pattern is seen in Fig.5.3, respectively. A single broad peak at $2\theta=23.9$, which corresponds to an intermediate layer distance of 0.83 nm, dominated the XRD pattern GO. The peak at $2\theta = 38.2$ disappearing indicates that the product has undergone chemical oxidation and is fully oxidised. The earlier studies found almost identical findings.

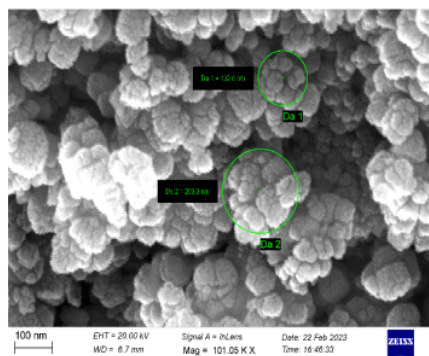
5.1.3. Fourier Transform Infrared Spectroscopy (FTIR).



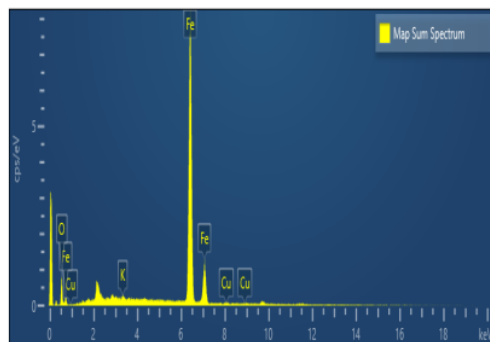
(fig.5.4)

Fourier Transform Infrared (FTIR) analysis is a well-established and trustworthy technique for analysing the molecular composition, functional groups, and chemical properties of materials, such as graphene oxide. The structure and properties of graphene oxide, a two-dimensional carbon-based material with unique properties and potential applications in a variety of fields, including materials science, nanotechnology, and biotechnology, are better understood thanks to FTIR analysis, which offers insightful information into the vibrational modes of molecules. In contrast, C=N stretching vibrations and skeletal motions from unoxidized graphitic domains are indicated by the bands at 1706 cm and 1041 cm, respectively (fig. 5.4). O-H stretching vibrations are represented by the strong band at 3294 cm. The band at 1035 cm suggests vibrations caused by C-O stretching. Additional characteristics include the peak at 1043 cm, which could be caused by the -C-O stretching frequency, and the peak at 1571 cm, which corresponds to the vibration spectrum of the carbon-carbon double bond. However, a second prominent peak that was seen at 1704 cm lends credence to the -C=N stretching frequency.

5.1.4. Scanning Electron microscopy (SEM) with EDX for Fe₃O₄/GO



(fig.5.5)



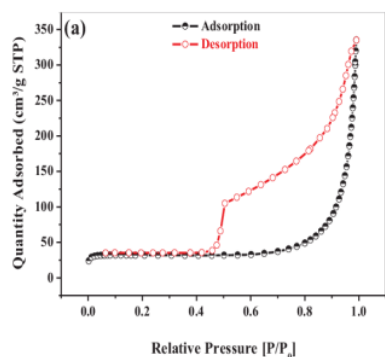
(fig.5.6)

For high-resolution imaging and elemental analysis of materials, scanning electron microscopy (SEM) in conjunction with energy-dispersive x-ray spectroscopy (EDX) is a particularly effective analytical method. Numerous domains, including materials science, nanotechnology, geology, forensic science, metallurgy, and others, find major usage for SEM with EDX. A finely focused electron beam analyses the sample surface during SEM with EDX analysis, creating secondary electrons, backscattered electrons, and unique X-rays. In-depth photos of the sample's surface morphology may be observed in the secondary and backscattered electrons, and the unique X-rays reveal essential data about the sample's elemental composition. Scanning electron microscopy was employed to investigate and characterise the synthesised nano-composite. Nanoparticle analysis is done using SEM. Figure 5.5 was scanned at a distance of 100 nanometers. The scanning electron microscope image makes it obvious that FeSO₄ was successfully attached to the graphene oxide, as evidenced by the porosity network that it generated, which has the appearance of a loose sponge. The nanocomposite's EDX graph is provided in (fig.5.6).

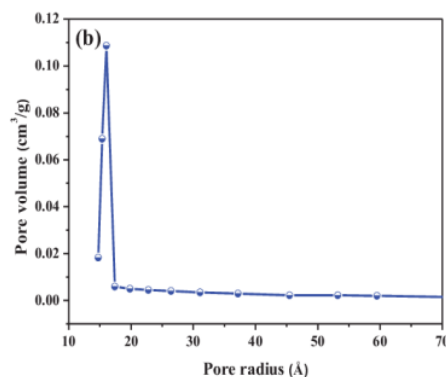
Element	Line Type	Weight %	Weight %Sigma	Atomic %
O	K series	17.05	0.20	32.18
K	K series	0.36	0.09	0.46
Fe	K series	67.91	0.34	61.2
C	K series	14.68	0.27	27.64
Total		100.0		100.0

(Table.5.1) EDX composition

5.1.5. Brunauer-Emmett-Teller analysis



(fig.5.7)



(fig.5.8)

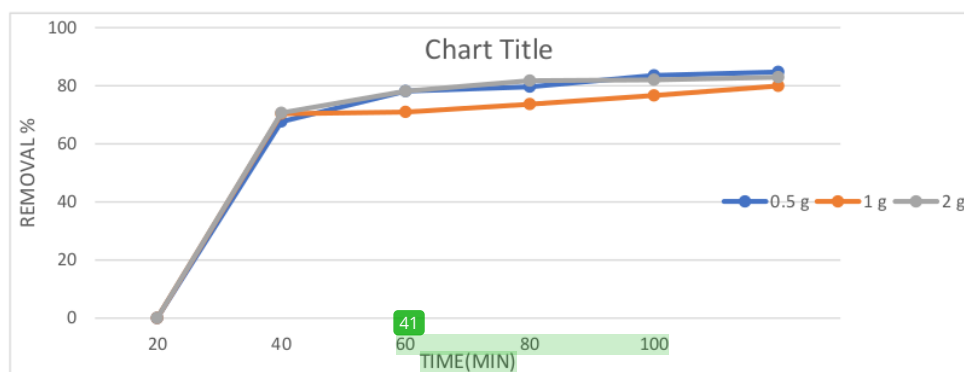
Brunauer-Emmett-Teller (BET) analysis is a well-liked and trustworthy technique for precisely calculating the surface area of materials, such as nanoparticles, porous materials, and catalysts. It is based on the concept of gas adsorption onto the sample surface and provides crucial details on the porosity, pore size distribution, and surface area of materials. BET analysis has applications in many fields, such as materials science, chemistry, chemical engineering, and environmental research, among others, since it provides an accurate estimate of surface area at the nanoscale. The total surface area of the synthesised graphene-based nickel nanocomposite was estimated using a Brunauer-Emmett-Teller (BET) surface area analyzer and nitrogen adsorption/desorption isotherms at a bath temperature of -196.3°C. The surface area was determined using the BET multi-point and single-point approaches based on the N₂ adsorption/desorption isotherm values. In order to clear the pores before analysis, the graphene-based nickel nanocomposite was vacuum evacuated for a whole night at 150 degrees Celsius. Using the BJH methodology, which integrates the DH method and Kelvin equation, the data on pore size distribution and pore volume were calculated. The BJH model was used to evaluate the pore size distribution, and the Dubinin-Radushkevich (DR) method was used to determine the micropore volumes of the samples. The BET surface area measurement parameters are summarised as follows: 11.0 m²/g of micropores, 5.9 nm pore size, 0.025 cm³/g of pore volume, and 16.1 m²/g of surface area.

5.2. EFFECT OF VARIABLES ON REMOVAL OF CHROMIUM VI

4

5.2.1. EFFECT OF NANO-COMPOSITE DOSAGE

The effect of adsorbent dosage on the adsorption process is a crucial factor that must be carefully considered. Adsorbent dosage refers to the quantity of adsorbent material used relative to the amount of adsorbate being treated. The optimal dosage of adsorbent has a important impact on the efficiency and effectiveness of this adsorption process. It is essential to accurately determine the appropriate dosage of adsorbent for a specific adsorption system, considering factors such as the characteristics of the adsorbate and adsorbent, desired level of adsorption, and operational parameters. To find out how the concentration affects the removal % of chromium, the range of concentration we use ranges from 100 ppm to 400 ppm, The dosages of 0.5g, 1.0g, 1.5g of weight were taken for consideration. The dosage for the below shown (fig.5.9) is 0.5g.



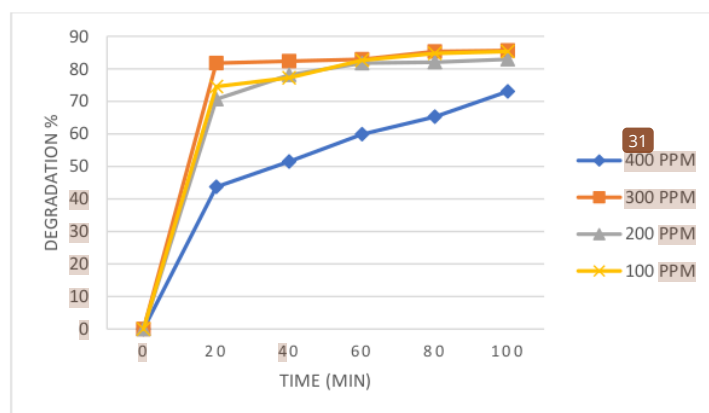
(fig.5.9)

5.2.2. EFFECT OF CONCENTRATION

23

A key factor in the adsorption process is the concentration of the adsorbate in the fluid phase. The impact of concentration is concerned with how the adsorbate's concentration affects the adsorbent material's capacity and effectiveness for adsorption. It is important to consider the impact of concentration when designing and optimizing adsorption processes. To find out how the concentration affects the removal % of chromium, the range of concentration we use

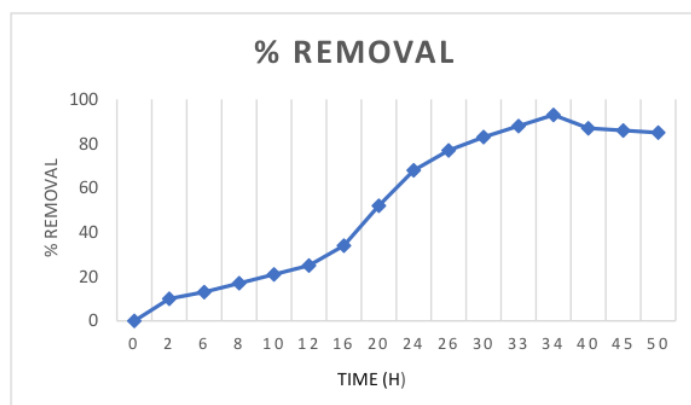
ranges from 100 ppm to 400 ppm, The dosages of 0.5g, 1.0g, 1.5g of weight were taken for consideration. The dosage for the below shown (fig.5.9) is 0.5g.



(fig.5.10)

5.2.3. EFFECT OF CONTACT TIME

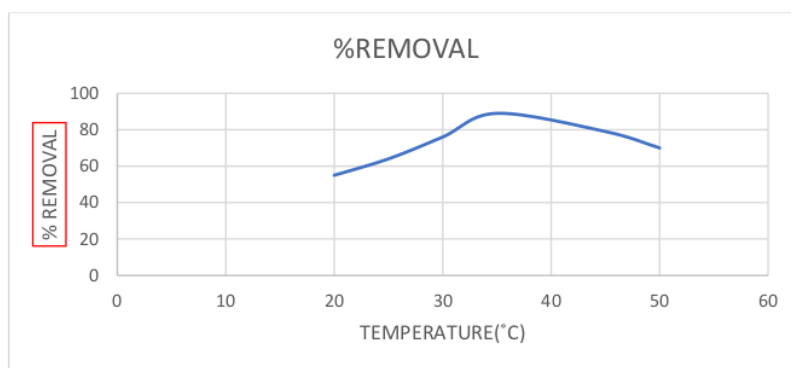
The effect of contact time on the adsorption process is crucial, since it impacts the duration that a chemical stay in contact with an adsorbent material. Several parameters, including the type of the adsorbate and adsorbent, temperature, concentration of the adsorbate in the fluid phase, and the desired amount of adsorption, play a critical role in defining the appropriate contact time for adsorption. The impact of contact time at the elimination of chromium VI is indicated in (fig.5.9). Removal efficiency improves as the duration of the procedure increases.



(fig.5.11)

14 5.2.4. EFFECT OF TEMPERATURE

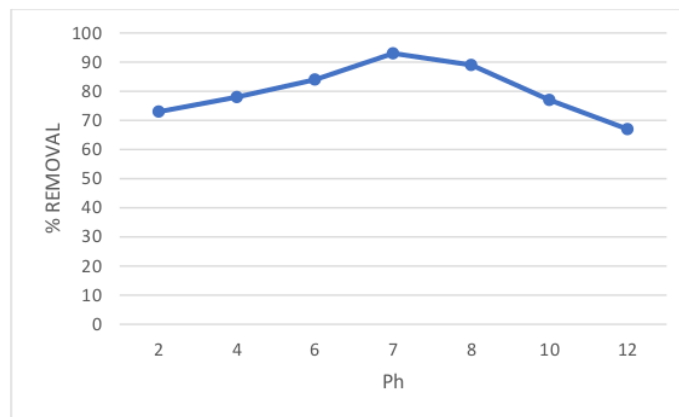
38 The influence of temperature on the adsorption process for chromium removal is important. Increase in temperature from 40°C to 50°C significantly affect the rate of adsorption owing to the greater kinetic energy of molecules, which promotes enhanced diffusion and mass transfer of chromium ions onto the adsorbent surface. However, if temperature is above 50°C results in decreased adsorption capacity due to changes in surface chemistry, pore structure. The below figure shows the % of removal as the temperature varies.



(fig.5.12)

11 5.2.5. EFFECT OF pH

24 The pH of the solutions plays a crucial role in the adsorption process for chromium removal. It disturbs the surface charge of the adsorbent material, the speciation of chromium ions, and the adsorption capacity and efficiency. It is important to consider the pH-dependent effects on these factors when designing chromium removal processes to ensure originality in the work done. The (fig.5.13) shown below impacts the pH removal on chromium when undergone through UV radiation. The chromium removal (%) determined with UV light for pH 2, 5, 6.5, 7.5, 10, and 11 were 73, 78, 84, 93, 89, and 77, respectively. So according to the below graph the optimal value of pH is found to be 7.5 as the maximum removal % is found to be 93%.



(fig.5.13)

5.3. ADSORPTION ISOTHERM

The distribution of an adsorbate, or the substance being adsorbed, between an adsorbent, or the substance enabling adsorption, and the solution phase at equilibrium, is described by adsorption isotherms, which are mathematical models. These isotherms are often used for modelling and analysis of adsorption processes in a variety of disciplines, including environmental science, chemical engineering, and materials science. They provide insightful information on the behaviour and properties of the adsorbent-adsorbate system. This experimental method makes it easier to determine the maximum adsorption capacity of the adsorbent, which depends on the mathematical model used. The models of Langmuir, Freundlich, and Temkin are some of the ones that are often used to describe solid-liquid sorption systems.

A frequently used model for examining chromium elimination by adsorption is the Langmuir isotherm. It presupposes that there are a limited number of identical adsorption sites on a homogeneous surface in a monolayer. The connection between the equilibrium adsorption capacity and the equilibrium concentration of the adsorbate in the solution is mathematically described by the Langmuir isotherm. On the other hand, the Freundlich isotherm is a popular adsorption model that is also utilised in the removal of chromium. On a heterogeneous surface, a multilayer adsorption with a variety of adsorption energies is assumed. The mathematical link between the adsorption capacity and the equilibrium concentration of the adsorbate is described by the Freundlich isotherm.

5.3.1. LANGMUIR ISOTHERM

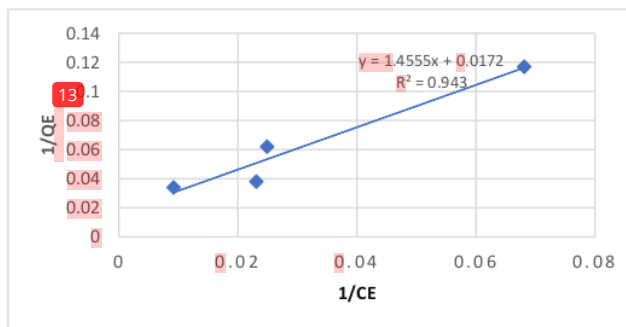
A common model in the study of adsorption is the Langmuir isotherm. Irving Langmuir first put out this theory in 1918, and it depicts the adsorption of a solute onto a solid surface as a monolayer with a limited number of adsorption sites. The Langmuir isotherm makes the assumptions that all adsorption sites have a constant adsorption energy and that the adsorption process is reversible.

Eqn3 is the Langmuir isotherm equation:

$$q = (q_m K L C) / (1 + K L C) \quad (3)$$

where:

q is the solute adsorbed quantity per unit mass of adsorbent, such as mg/g. The adsorbent's maximal adsorption capacity is q_m (ex. mg/g). Langmuir adsorption constant (L/mg) is KL . c is the solute's equilibrium concentration in the solution (example: mg/L).



(fig.5.16)

5.3.2. FREUNDLICH ISOTHERM

The Freundlich isotherm, proposed by Herbert Freundlich in 1906, is a commonly employed model in this field of adsorption. It describes the adsorption of solutes onto a solid surface with multiple layers of adsorption and assumes a heterogeneous surface with varying adsorption energies for different adsorption sites.

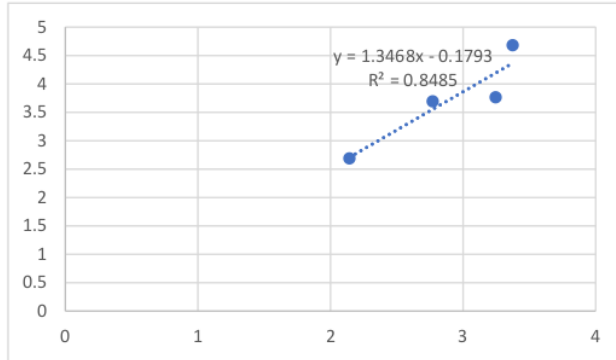
The Freundlich isotherm equation is given by Eqn4:

$$q = K F C^{(1/n)} \quad (4)$$

9

where:

q is the solute adsorbed quantity per unit mass of adsorbent, such as mg/g. Freundlich adsorption constant (mg/g) / (mg/L)^(1/n) is known as KF. c is the solute's equilibrium concentration in the solution (example: mg/L). The Freundlich exponent (n) characterises the form of the isotherm and symbolises the variability of the adsorption process.



(fig.5.17)

5.3.3 TEMPKIN ISOTHERM

The Tempkin isotherm, originally proposed by M. Tempkin and V. Pyzhev in 1940, is a widely employed model in adsorption studies that accounts for the impact of adsorbate-adsorbent interactions and surface heterogeneity. This model based on the assumption that the heat of the adsorption diminishes linearly with the coverage of adsorbate on the adsorbent surface.

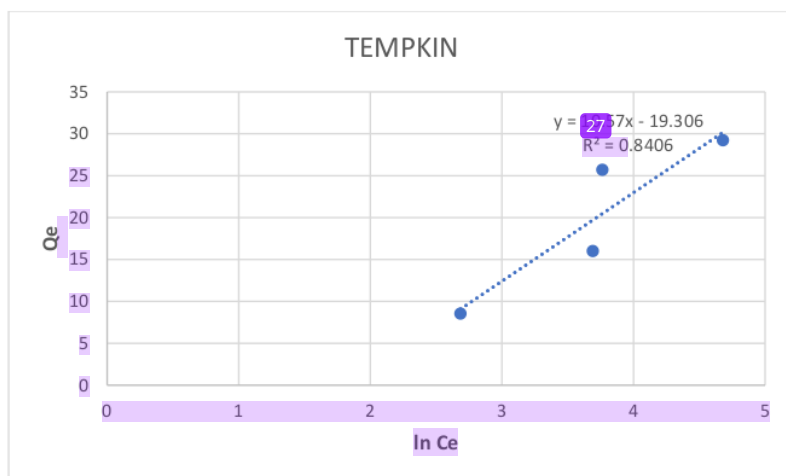
The Tempkin isotherm equation is given by Eqn 5:

$$q = B \ln(At ka + 1/B) \quad (5)$$

9

where:

q is the solute adsorbed quantity per unit mass of adsorbent, such as mg/g. The heat of adsorption (J/mol)-related Tempkin isotherm constant is B. The equilibrium binding constant (L/g) is called At. ka is the adsorption capacity (g/L)-related equilibrium constant.



(fig5.18)

MODELS	Isothermal Constant			
	q_e (mg/g)	K_L (L/mg)	R_L	R^2
Langmuir	42.61	0.015	0.05–0.35	0.943
Freundlich	n	(mg/g)	-	R^2
	2.463	5.69	-	0.848
Tempkin	(L/g)	(mg/L)	(J/mg)	R^2
	0.53	6.78	274.66	0.840

(Table5.2) ADSORPTION ISOTHERMS

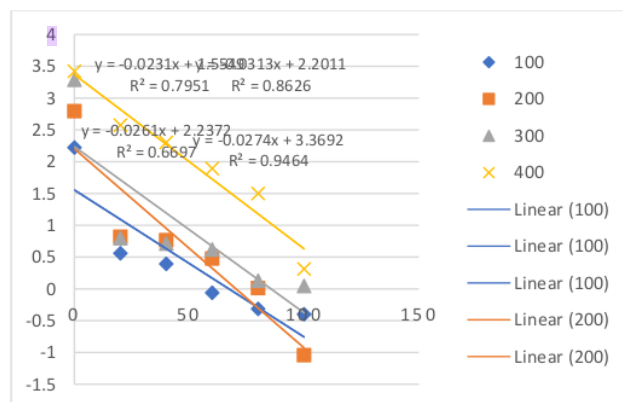
5.4. KINETIC STUDIES OF ADSORPTION

Adsorption kinetic studies are crucial for determining how quickly a material clings to the surface of a solid or liquid, producing a layer. Adsorption plays a crucial role in various scientific and industrial applications, such as catalysis, environmental remediation, and drug delivery. These studies typically involve measuring changes in the concentration or coverage of the adsorbate on the adsorbent over time. This experimental data obtained from these

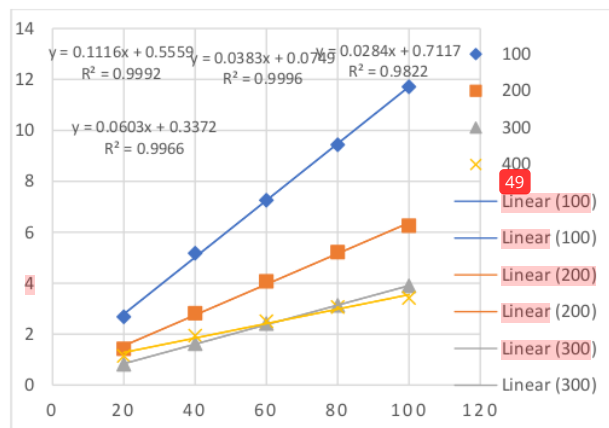
studies are then analyzed to determine the rate of adsorption and the underlying mechanisms, providing insights into the nature of this adsorption process and the factors that affect it. By understanding the kinetics of adsorption, we can optimize adsorption processes, design efficient adsorbents, and predict the performance of adsorption systems under different conditions.

5.4.1. PSEUDO FIRST & SECOND ORDER REACTION

The experimental data for the adsorption of chromium (VI) were analysed in this work using both the pseudo-first-order and pseudo-second-order models (figs. 5.14, 15). The pseudo-first-order and pseudo-second-order figures are provided below. The adsorption of chromium (VI) was shown to follow a pseudo-second-order kinetic model after comprehensive investigation of the kinetic data. The pseudo-second-order model gave a better match to the experimental data than the pseudo-first-order model, according to a comparison of the kinetic constants derived from the pseudo-first-order and pseudo-second-order graphs. The rate-limiting stage of the adsorption process may entail chemisorption or strong chemical interactions between the adsorbate and adsorbent, according to this suggestion that the adsorption of chromium (VI) is better represented by a pseudo-second-order kinetic model.



(fig5.14)



(fig.5.15)

5.4.1.1 Kinetics of Chromium VI adsorption

Pseudo 1 st order	k_1 (min ⁻¹)	q_e (mg/g)	R^2
	0.0214	67.56	0.946
Pseudo 2 nd order	k_2 (min ⁻¹)	q_e (mg/g)	R^2
	0.00042	58.21	0.994

(Table.5.2)

5.5. MECHANISM OF ADSORPTION

With positively charged chromium ions (Cr) in aqueous solutions, often in the form of Cr(VI) or Cr(III), graphene oxide (GO), which has a negatively charged surface due to the presence of oxygen functional groups, may exchange ions with them. Since the positively charged Cr ions and the negatively charged surface of GO have a high electrostatic affinity, this results in the substitution of surface oxygen functional groups with Cr ions. Diverse oxygen functional

⁴ groups, such as hydroxyl (OH), epoxy (C-O-C), and carboxyl (COOH) groups, are abundant on the surface of graphene oxide. These groups promote powerful interactions with chromium ions (Cr) through ⁵¹electrostatic attractions, hydrogen bonds, and ⁶¹van der Waals forces. Therefore, via these interactions, Cr ions may adsorb onto the surface of graphene oxide, leading to the creation of Cr-GO surface complexes. Both graphene oxide (GO) and Fe₃O₄ nanoparticles include oxygen functional groups that may form chemical bonds with chromium (Cr) ions by coordination or chelation, leading to chemical adsorption. The Fe₃O₄/GO composite's surface oxygen functional groups and the Cr ions may form complexes, which effectively removes the Cr ions from the solution.

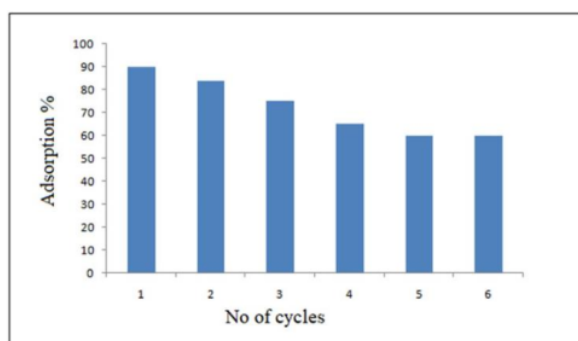
5.6. COMPARITIVE STUDIES

⁴ Adsorbent	Concentration of absorbent (g L ⁻¹)	pH	Contact time (min)	Adsorption capacity (mg g ⁻¹)	Ref.
Layered zinc hydroxide nitrate	1.5	8	120	194.3	32
⁴ Amine-based-surfactant-modified clinoptilite	14	4	120	6.72	34
Superporous magnetic cationic cryogels	0.15	7	180	65.5	35
Magnetic ionic liquid/chitosan/graphene oxide	1.0	3	40	145.35	36
HDTMA-zeolite	25	3	240	8.83	6
Nickel ferrite composite built on graphene oxide	2.5	4	120	44.9	45
Fe ₃ O ₄ /graphene nanocomposites	1.0	7.5	90	85.6	THIS WORK

(Table.5.3) comparison of different adsorbents

5.7. REGENERATION STUDIES

In this work, we showed that the synthesised graphene oxide/Fe₃O₄ nanocomposite for Cr(VI) adsorption is regenerable and reusable. Initially introduced to a beaker, the adsorbent was given 60 minutes to absorb Cr (VI) while being vigorously stirred. After that, deionized water was used to filter and clean the adsorbent. In order to activate the adsorbent sites and dissolve all adsorbed metal, the utilised adsorbent was heated to around 150°C. The filtrate's absorbance was measured after this cycle was run six times, with each round including 60 minutes of stirring with Cr (VI) metal. The outcomes, as seen in Figure 9, were encouraging. The efficacy of the adsorbent dropped to 85% after the first recycling, which might be due to nanoparticle clustering during sintering. However, the efficiency continued to decline in the same sequence for the second round and following cycles. Despite this, the substance may still be employed for many iterations of adsorption or purification, demonstrating its capacity for regeneration and reusability. In conclusion, our research showed that Fe₃O₄/GO nanocomposite may be recycled and utilised again for Cr(VI) adsorption, however the effectiveness may somewhat deteriorate after many cycles. The adsorbent is a desirable solution for affordable and environmentally responsible wastewater treatment and other adsorption applications due to its regenerable nature.



(fig5.16)

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