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MSCHE-F21-002

FOS

Removal of Heavy Metals from Industrial Drains by Using Iron Based Nano Composite



SUPERIOR UNIVERSITY

Thesis Submitted to

The Superior University Lahore

In Partial Fulfillment of the

Requirement for the Degree of

M. Phil. Chemistry

By

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I hereby state that my MPhil thesis titled “**Removal of heavy metals from industrial drains by using iron based nano composite**” is my work and has not been submitted previously by me for taking any degree from this University,

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No part of this thesis has been submitted anywhere else for any other degree. This thesis is submitted to the Faculty of Sciences, The Superior University, Lahore in partial fulfillment of the requirements for the degree of Master of Science/Master of Philosophy in the field of “**Chemistry**” in Faculty of Sciences at The Superior University, Lahore.

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Dedication

I dedicate this thesis to

MY FATHER

For earning an honest living for us and for supporting and
encouraging me to believe in myself

MY MOTHER

A strong and gentle soul who taught me to trust ALLAH,
believe in hard work and that so much could be done with little

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In the name of ALLAH, the most Gracious, the most Merciful. All praises to ALLAH and His blessings for the completion of this thesis.

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List of Abbreviations

AAS	Atomic Absorption Spectroscopy
BET	Brunauer-Emmett-Teller
PPy	Polypyrrole
RSM	Response Surface Methodology
SEM	Scanning Electron Microscopy
SPIONs	Super paramagnetic Iron Oxide Nanoparticles
TDS	Total Dissolved Solids
TGA	Thermo gravimetric Analysis
UV-Vis	Ultraviolet-Visible Spectroscopy
XRD	X-Ray Diffraction
EPA	Environmental Protection Agency
FTIR	Fourier Transform Infrared Spectroscopy
GO	Graphene Oxide
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
LDH	Layered Double Hydroxide
MB	Methylene Blue
MgO	Magnesium Oxide
MnO ₂	Manganese Dioxide
MO	Methyl Orange
Ni	Nickel
Pb	Lead
Cd	Cadmium
Cr	Chromium
Cu	Copper
Fe	Iron

ABSTRACT

Contaminated water with heavy metals poses a significant risk to both the environment and public health. In this study, iron and magnesium oxide nano composites were synthesized and utilized to efficiently remove heavy metals from industrial wastewater. Iron based nano composites were synthesized by the precipitation-calcination method which has a mesoporous structure with high surface area and significant adsorption property. Characterization techniques such as Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), and Fourier Transform Infrared Spectroscopy (FTIR) techniques that confirmed successful integration of iron and magnesium oxide, with the nano composites exhibiting strong adsorption capabilities for cadmium (Cd) ions. The iron based nano composites removed heavy metals with the efficiencies as high as 97.73% under optimal operating conditions, as demonstrated by adsorption experiments. The magnetic qualities of the nano composites made it easy to recover and reuse, thus being a cost-effective solution for treating wastewater. With its focus on eco-friendly materials and approaches, the study has significant implications for environmental remediation efforts that can ultimately protect public health from the serious effects of water pollution.

CHAPTER 1

INTRODUCTION

All living organisms existing on the earth whether plants or animals need water for survival. The primary issue is the contamination of drinking water by chemicals and heavy metals arising from different sources [1]. Ground water is used for industrial, agricultural and daily water needs by the people all over the world [2]. However, the World Health Organization (WHO) reported that 80% of diseases worldwide are due to inadequate sanitation facilities, and contaminated drinking or lack of availability [3]. Highly populated and Industrial areas are having serious critical problems with the purity of the water [4]. Some other dyes and heavy metals released into the water from various industries, particularly the textile industry, are hazardous to human organs like kidney, liver and brain due to its chemical substances [5].

One third of the world population usually uses groundwater for drinking [6]. Water becomes polluted simply by means of its usage all activities, whether household, agricultural and industrial activity generates waste water with unwanted and sometimes toxic contaminants [7]. Currently, around 80% of people in urban areas and about 11% rural people have the right to access the piped water supply [8].

Polluted water by toxic metals can cause various serious diseases, the acute short-term (e.g., causing diarrhea), and the chronic (e.g., causing deaths) [9]. Around 40% of the population does not even have hygienic facilities, while majority does not have access to drinking water [10]. Millions of people, including children, killed due to unclean water and poor sanitation every year [11]. Approximately 6,000 children die from poor sanitation, bad hygiene and a lack of clean water. Currently, approximately 80% of the people in urban places and 11% in rural places have a right to the piped water supply [12].

In fact, 2.2 million people die every year due to the crisis, 6000 children die every day due to unhealthy water and poor hygiene [13]. Poor water quality is an everyday reality for most people in many emerging countries. By 2025, half the people in 52 countries will face severe water crises, especially in South Asia, where freshwater sources are depleted and pollution is on the rise [14]. Most of heavy metals are harmful or toxic which may harm the environment or the human body. Heavy metal pollutants found in wastewater are Mercury (Hg), copper (Cu), nickel (Ni), lead (Pb)

chromium (Cr) cadmium (Cd), zinc(Zn) and iron(Fe)[15]. Heavy metals are released through a wide range of processes, which include combustion (of fuels), transport (free dust) and disposal into air, water and soil [16].

Heavy metal pollution such as lead (Pb), cadmium (Cd), copper (Cu), and iron (Fe) in the wastewater increased significantly over the past few years [17]. The rise is directly connected to the usage of electronic devices, notably personal gadgets like cell phones and tablets [18]. As these devices becoming more prevalent, they are producing waste containing heavy metals that can contaminate water sources when improperly disposed of [19].

Heavy metals like lead, mercury, cadmium, and arsenic are harmful and have adverse effects on the environment [20]. Once they enter the ecosystems, they tend to pollute the soil, water, and air, thus disturbing the ecosystem balance and also posing a major threat to plants, animals, and humans. Unlike the pollutants which are degraded with time, heavy metals are non-degradable and they can accumulate in the environment causing chronically damage [21].

In water, heavy metals can build up in the bodies of aquatic life. These metals increase in concentration as they pass through the food chain and ultimately threaten larger predators and human populations who consume these animals [22]. These metals, however, can disrupt natural processes in soil, preventing plant growth and inhibiting soil bacteria, which aid in the maintenance of ecological balance [23]. They can also cause a wide variety of health problems in animals and humans, including neurological and developmental disorders [24].

Various methods are already being employed for the solution of problem of water such as membrane filtration, ion exchange, coagulation, adsorption, chemical precipitation and biological treatment to tackle it [25]. The most commonly applied of such strategies was adsorption, a direct, highly efficient, and inexpensive approach [26]. Bio- and carbonaceous adsorbents such as chitosan, carbon nanotubes (CNTs) and graphene oxide have great selectivity for the adsorption but they are usually related with high-cost of production [27]. Metal oxides and silica If used as a highly thermally stable support material, it must be considered that these oxide adsorbents such as metal oxides and silica have higher [28].

In particular, iron-based nanoparticles (Fe-NPs) are a type of nanotechnology that have potential in overcoming the problems caused by wastewater and heavy metals released from industries [29]. Fe-NPs are highly reactive and therefore can absorb

large amounts of toxins present in wastewater, such as heavy metals, dyes and organic compound [30]. Iron based nanocomposites in batch adsorption is one potential new innovative treatment for the issue caused by the wastewater and heavy metals emitted from industries. These are inexpensive, polymer-based Fe-NPs, which have exhibited high efficiency and an easy execution, a matter that makes them a suitable solution to wastewater treatment [31].

This study focusses on addressing an important environmental pollution organic waste cause by industrial practices through development on iron and magnesium based nano composites [32]. The iron and magnesium based nano composites are considered as low cost, high efficiency and easy synthesis candidates for wastewater treatments [33] Results of the study are considered to be acceptable for the efficient removal of heavy metals from drain water.

AIMS AND OBJECTIVES

The aim of this research is to control cadmium pollution in industrial wastewater drains, aiming to improve water disposal safety and public health. The primary objective of this study is to synthesize and trial a nanocomposite material designed for effective cadmium removal. To achieve this goal, the following specific objectives have been established:

1. Synthesize iron-based nanocomposite through co-precipitation and calcination techniques.
2. Collect wastewater samples from a local industrial drain.
3. Determine cadmium levels in the wastewater through Atomic Absorption Spectroscopy (AAS).
4. Assess the adsorption capacity of iron-based nanocomposite for cadmium removal across various conditions.
5. Analyze the synthesized nanocomposite using SEM, FTIR, and XRD methods.

CHAPTER 2

LITERATURE REVIEW

This study focuses on the synthesis of magnetic iron and magnesium oxide nano composites using a simple precipitation-calcination method, aimed at evaluating their effectiveness in removing heavy metal ions and dyes from water [34]. The pre-arranged iron and magnesium oxide center shell nano composites showed a mesoporous structure, enormous surface region, and high attractive immersion esteem, working with attractive detachment [35]. The iron based nano composites exhibited exceptional adsorption properties, with Pb (II) and methyl orange (MO) having maximum adsorption capacities of 1476.4 mg⁻¹ and 6947.9 mg⁻¹, respectively [36]. The adsorption cycles of Pb (II) and MO on Fe@MgO followed the pseudo-second-request dynamic model and Langmuir isotherm model, individually, showing monolayer chemisorption ways of behaving [37]. The components of Pb (II) and MO adsorption were additionally examined by investigating the adsorbed items. It was observed that a limited quantity of MO was mineralized by iron and magnesium. In addition, iron based nanocomposites showed synchronous expulsion of pb (II) and MO from water, with Pb (II) advancing the MO adsorption onto iron and magnesium oxide [38]. These discoveries recommend that iron based nanocomposites hold critical potential for natural remediation, especially for eliminating weighty metal particles and colors from water [39].

In situ polymerization of pyrrole monomer was organized to remove extremely poisonous Cr (VI) nanocomposite of polypyrrole (PPy)/Fe₃O₄ [40]. The characterization of the nanocomposite was performed with several methods including ATR-FTIR, X-ray diffraction (XRD), FE-SEM, and HR-TEM to investigate the structure and morphology [40]. The presence of magnetic nature of the nanocomposite was tested through ESR measurements [41].

Up to 100 % of Cr (VI) was adsorbed from 200 mg/L liquid solution by the nanocomposite at pH 2. The adsorption of Cr (VI) on the surface of nanocomposite was corroborated by ATR-FTIR and X-ray photoelectron spectroscopy (XPS) [42]. Furthermore, XPS focuses on suggesting that ion exchange and reduction processes might be responsible for Cr (VI) removal by the PPy/Fe₃O₄ nanocomposite [43]. The nanocomposite's adsorption productivity for Cr (VI) diminished with an expansion in PH [44]. The pseudo-second-request rate model best depicted the adsorption energy,

while the Langmuir isotherm model gave a solid match to the isotherm information [45]. A thermodynamic report uncovered that the adsorption cycle is unconstrained and endothermic in nature. In spite of the low recuperation of adsorbed Cr (VI), the recovered adsorbent was viewed as reusable for two progressive adsorption-desorption cycles with next to no critical loss of its unique limit [46].

Alumina Nano Composite delivered by joining the adsorption properties of nano-actuated alumina with the attractive elements of iron oxides [47]. This brought about the production of a nano-attractive adsorbent that can be effectively isolated from the medium by utilizing a straightforward attractive interaction after adsorption [48]. The extensive portrayal of MANC utilizing various strategies like XRD, SEM, TEM, EDX, and surface region (BET). Then utilized a Quantum Plan SQUID magnetometer to quantify the attractive properties [49]. The essential goal was to survey the adequacy of MANC in eliminating cadmium particles from watery arrangements through group adsorption. the impacts of a few factors, was analyzed for example, pH, adsorbent dose, temperature, contact time, and starting Cd^{2+} focus on the adsorption of cadmium particles [50]. Langmuir, Freundlich, and Temkin isotherms utilized to fit the harmony information, and observed that the Langmuir isotherm was the best portrayal [51]. To examine the dynamic information, we utilized pseudo-first-request, pseudo-second-request, Elvish, and intraparticle dissemination models. It found that pseudo-second-request model gave the best fit [52]. The thermodynamic boundaries determined for the Cd^{2+} particle MANC framework, and established that the adsorption cycle was endothermic in nature, as demonstrated by the positive worth of ΔH [53]. In light of the harmony information got, we planned a solitary stage bunch absorber for the evacuation of Cd^{2+} particles by MANC [54]. These discoveries exhibit that MANC can possibly be a powerful adsorbent for the expulsion of cadmium particles from watery arrangements [55].

In specific examination, researchers effectively synthesized a progressive nanocomposite comprising of a magnetic core of Fe_3O_4 coated with a amorphous MnO_2 shell utilizing a gentle hydrothermal process [56]. The resulting $\text{Fe}_3\text{O}_4/\text{MnO}_2$ nanocomposite showed a particular flowerlike morphology, furnishing it with a significant surface region and high magnetic saturation value [57]. These properties guaranteed excellent sorption abilities and easy separation.

The researcher studied the application of $\text{Fe}_3\text{O}_4/\text{MnO}_2$ for eliminating heavy metal ions from water containing Cd (II), Cu (II), Pb (II), and Zn (II). Comparative analyses

indicated that $\text{Fe}_3\text{O}_4/\text{MnO}_2$ exhibited significantly enhanced removal efficiency compared to unaltered Fe_3O_4 nanoparticles [58]. This enhancement is attributed to the exceptional properties of the MnO_2 coating.

Further examination focused on the adsorption behavior of $\text{Fe}_3\text{O}_4/\text{MnO}_2$ towards Cd (II) [59]. The equilibrium information data were effectively described by the Langmuir model, with a most extreme adsorption limit of 53.2 mg g^{-1} noticed for Cd (II). Significantly, $\text{Fe}_3\text{O}_4/\text{MnO}_2$ exhibited remarkable stability, holding more than 80% of its adsorption limit under different conditions typical of natural waters [60].

The nanocomposite additionally showed brilliant recyclability and reusability. Through continuous adsorption-desorption tests supported by an external magnetic field, $\text{Fe}_3\text{O}_4/\text{MnO}_2$ could be easily recovered and reused without any loss in its adsorption limit [61].

In summary, the work suggests $\text{Fe}_3\text{O}_4/\text{MnO}_2$ as a potential and efficient adsorbent to remove heavy metal ions in polluted water systems [62]. Its good hierarchical structure, high adsorption capacity, stability and regeneration enable it to become the most potential feasible candidate for sustainable applications in environmental and water treatment [63].

The purpose of this work was to prepare a nano-hybrid of $\text{MgO}/\text{Fe}_3\text{O}_4$ for chromium removal from water by reducing the Cr (VI) ions or dichromate anions ($\text{Cr}_2\text{O}_7^{2-}$) first [64]. In order to maximize the chromium removal efficiency, the specialists attempted to isolate through which contact time, nano-composite weight, Cr (VI) concentration, temperature and pH were optimum operating conditions. The $\text{MgO}/\text{Fe}_3\text{O}_4$ nanocomposite was analyzed using BET, VSM, SEM, TEM, FTIR, XRD and EDAX techniques [65].

Furthermore, VSM investigational also means that Fe_3O_4 nanoparticles confirmed super paramagnetic assets withinside the researches of Fe_3O_4 and $\text{MgO}/\text{Fe}_3\text{O}_4$ nanocomposites [60]. Specific surface area of the nanocomposite showed $75.21 \text{ m}^2 \text{ g}^{-1}$ by BET analysis [66].

The adsorption study exhibited that the best operating parameters for removal of Cr (VI) ions with the use of the $\text{MgO}/\text{Fe}_3\text{O}_4$ nanocomposite were pH 3, at a contact time of 30 min, temperature at 25°C , initial concentration of chromium ions 10 mg L^{-1} and conducted at an amount of adsorbent weight equal to 1.5 g L^{-1} . So apparently were able to act like the other 97.73% of people in these circumstances [67].

Furthermore, equilibrium, kinetics, and thermodynamics studies were carried out to

explain the adsorption cycle of Cr (VI) particles. The adsorption was determined to be well expressed by a pseudo-second order model. Both Langmuir and Freundlich models were found to properly represent the equilibrium [68]. Based on Langmuir model, the maximum adsorption capacity was 23.9 mg g⁻¹. Thermodynamic study showed a spontaneous and endothermic nature of the removal of Cr (VI) by 5MgO/Fe₃O₄ nanocomposite [69].

The present work could successfully integrate an MgO/Fe₃O₄ nanocomposite for Cr (VI) decontamination from aqueous medium. Optimum operational conditions were identified and the adsorption cycle was analyzed in equilibrium, energy and thermodynamic aspects. The findings indicated that the MgO/Fe₃O₄ nanocomposite was effective for chromium adsorption [70].

Heavy metal pollution of water also has emerged as a serious global issue, emphasizing the urgent need for efficient and adaptable water treatment methods. Recently, an increasing attention has been directed to actually Superparamagnetic iron oxide nanoparticles (SPIONs) and the composites with these as adsorbents of the heavy metallic ions via aquas system (69). The effective audit meta examination will be conducted which aims to collect and analyze the adsorption performance of SPION based nano adsorbents for heavy metals, in particular, lead, mercury, chromium, copper, arsenic, nickel and cadmium between 2011 to 2020 [71].

A far-reaching screening and examination of 232 articles recognized them as reasonable for the survey segment. Moreover, 180 distributed investigations met the quantifiable standards for the meta-examination. Computer programming language was used for, heterogeneity analysis, and the development of forest plots. Subgroup examination was conduct for each heavy metal ion to understand the source of variation in the data [72].

The aggregate information showed adsorption limits going from 0.23 to 1476.4 mg/g. The systematic survey of the examinations showed that the expulsion effectiveness of heavy metal particles by SPIONs changed somewhere in the range of 50 and 100 percent. In addition, the pH scope of 4 to 7 was the most broadly explored range for the removal of heavy metal ions. Adsorption tests were performed from 1 to 2500 minutes [73].

This article underlines the significance of achieving greatest removal effectiveness of metal particles in water treatment processes and examines the connection between adsorption dose and removal efficiency. The examination of the distributed

information ($k = 258$) revealed an I2 worth of 79% with regards to pooled removal efficiency information, recommending no distribution inclination [74].

This precise survey and meta-examination give important experiences into the adsorption execution of SPION-based nano adsorbents for heavy metal particles in aqueous system. The finding highlights the meaning of advancing expulsion effectiveness in water treatment and features the requirement for additional examinations in this field. The statistical examination shows the shortfall of publication bias, guaranteeing the believability of the ends drawn from the compiled data [75].

The primary goal of this study was to create MgO/Fe₃O₄ nanocomposite to remove Cr (VI) ions or anion dichromate from aqueous media and to identify the best operating conditions, including the impact of pH, temperature, contact time, nanocomposite concentration, and Cr (VI) concentration in aqueous solution. The MgO/Fe₃O₄ nanocomposite was characterized using a variety of analyses, including BET, VSM, SEM, TEM, FTIR, XRD, and EDAX/MAP. VSM analysis of Fe₃O₄ and MgO/Fe₃O₄ nanocomposite showed superparamagnetic nature of the Fe₃O₄ nanoparticles. Furthermore, the BET analysis showed specific surface area of 75.21 m²/g [76]. The adsorption isotherms data also showed that the optimum conditions for removing Cr (VI) ion from MgO/Fe₃O₄ nanocomposite at pH 3 and contact time, temperature, initial chromium concentration and adsorbent dose. This time, the elimination ratio was 97.73%. The Cr (VI) adsorption system was further studied for equilibrium, kinetic and thermodynamic studie. Moreover, The Freundlich and Langmuir adsorption isotherms could generate the experimental equilibrium data. The Langmuir model revealed that a total maximum adsorption capacity of 23.9 mg g⁻¹ was obtained. In addition, thermodynamics study was performed and coordinates with exothermic removal of chromium (VI) with MgO/Fe₃O₄ nanocomposite [77].

The as-prepared pollen-morphic MgFe₂O₄/Fe₂O₃ featured a hierarchically porous structure consisting of 8 nm-sized nano crystallites. The adsorption behavior of Congo red (CR) and minocycline (MC) on MgFe₃O₄/Fe₂O₃ was investigated from kinetic, equilibrium, and thermodynamic aspects. The magnetic microspheres once synthesised displayed excellent adsorption performance. The maximum adsorption capacity of Congo red and minocycline in the developed ISs were 259.1 mg/g and 200.8 mg/g, respectively. Both the Congo red and minocycline adsorption processes were spontaneous and endothermic in nature, which was well defined by pseudo-

second order and Langmuir monolayer models on as-produced samples, respectively. The adsorbed samples could be readily magnetically removed from the aqueous solution and rejuvenated with methanol or NaOH solution. The materials created through synthetic means may function well as adsorbents for environmental processes [78].

In this study, magnetic heavy metal removal from water was successfully achieved by easy hydrothermal synthesis of magnetic $\text{Fe}_3\text{O}_4\text{-MnO}_2$ nanoplates. The Fe_3O_4 component made it easier to recycle the adsorbent. As a result of the addition of amorphous MnO_2 , $\text{Fe}_3\text{O}_4\text{-MnO}_2$'s specific surface area was greatly increased, and the point of zero charge was lowered, ensuring strong metal cation adsorption capability. The effects of pH on removal efficiency and the isotherms and kinetics of the five metal ions on $\text{Fe}_3\text{O}_4\text{-MnO}_2$ were carefully examined. Sips isotherm was found to have the best match for Ni^{2+} , while Temkin, Redlich-Peterson, and Langmuir models had the best fit for Pb^{2+} , Cu^{2+} , Cd^{2+} and Zn^{2+} adsorption on $\text{Fe}_3\text{O}_4\text{-MnO}_2$. Pb^{2+} , Cu^{2+} , Cd^{2+} , Zn^{2+} and Ni^{2+} have adsorption capabilities of 208.17, 111.90, 169.90, 100.24, and 55.63 mg/g on $\text{Fe}_3\text{O}_4\text{-MnO}_2$, respectively. The pseudo-second-order model provided a better match to the kinetic data ($R^2 > 0.99$), showing that chemisorption played a major role. Ion exchange and complexation were mostly used to produce the adsorption process, according to further study of infrared spectra. The order of the metal ions' adsorption rates and preferences on $\text{Fe}_3\text{O}_4\text{-MnO}_2$ was Pb^{2+} , Cu^{2+} , Cd^{2+} , Zn^{2+} and Ni^{2+} [79]. Additionally, the adsorbent demonstrated strong surface protonation and deprotonation abilities as well as good recyclability when it came to neutralizing solutions. The results presented here have broad implications for the actual removal of metal cations from water [80].

Magnetic metal-organic framework (MOF) based nanocomposite material, termed $\text{ZIF-8@SiO}_2\text{@MnFe}_2\text{O}_4$ was successfully prepared and applied. Here, a microporous magnetic nanocomposite (MMNC) was prepared to adsorb Malachite Green (MG) and Methyl Orange (MO) dyes at the same time as well as under sonication. The surface and structural properties of the adsorbent were evaluated by Fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) with EDX, X-ray diffraction analysis (XRD), Brunauer-Emmett-Teller (BET), and vibrating sample magnetometer (VSM). The zero-order and first-derivative spectrophotometric techniques were utilized to quantify dye concentrations in binary room temperature solutions. Process variables

were optimised using response surface methodology (RSM) to study the individual as well as interactive impacts of the parameters. Finally, the optimal parameters were determined as 15 min contact time, 0.005 g adsorbent dosage and the dye concentrations of 13 mg L⁻¹ for MG and 10 mg L⁻¹ for MO. Removal efficiencies of MG and MO, under these conditions, were about 92.5% and <6 %, respectively. The adsorption data was well fitted to both Langmuir isotherm and pseudo-second-order kinetic models. The values of the maximum uptake capacities estimated were 1010.2 mg g⁻¹ for MG and 78.12 mg g⁻¹ for MO after reaching equilibrium, additionally, good magnetic property allow efficiently to selectively remove MG from MG/MO mixture by an external magnet and can be recycled repeatedly without losing adsorption efficiency. [81].

The primary goal of this study is to develop cost-effective, high-performance magnetic multilayer nanocomposites that can efficiently eliminate dyes from wastewater. The focus is on ensuring easy separation and long-term reusability. To achieve this, Polyethylene glycol (PEG) was utilized to modify the surfaces of Fe₃O₄ particles in combination with magnesium–aluminium layered double hydroxides (MAN–LDH) of varying molecular weights. The materials were analyzed using several techniques including inductively coupled plasma optical emission spectroscopy (ICP–OES), scanning electron microscopy (SEM), transmission electron microscopy (TEM), energy-dispersive X-ray spectroscopy (EDX), thermogravimetric analysis (TGA), and FTIR. The success of the optimized composite (6FPL) was attributed to its ability to carry out rapid nitrate ion exchange within the MAN–LDH interlayer structure, increase surface area, and establish strong hydrogen bonding and electrostatic attraction with dye molecules. Thermodynamic evaluations indicated that the process was spontaneous and endothermic. Furthermore, all FPL composites exhibited outstanding regeneration capabilities, with MAN–LDH-coated materials maintaining full adsorption efficiency even after 31 reuse cycles, equivalent to freshly prepared 6FPL. [82].

A hierarchical core–shell nanorods composed of magnetic iron oxide cores wrapped by layered magnesium silicate shells is prepared through a sol–gel process in another route. These HIO@MgSi nanorods are mainly well-dispersed ultra-thin magnesium silicate (ca. 5 nm thick) nanosheets surrounding the Fe₃O₄ cores. The results of the using methylene blue as a model contaminant show very high removal efficiency (99.3%) upon 40 minutes and it reached a maximum adsorption capacity of 2020.20

mg g⁻¹ after 4 hours; these results are better than those of any COC up to this date. HIO@MgSi nanorods provide a new direction for the rapid and highly efficient purification of cationic organic pollutants in industrial effluents and drinking water sources [83].

Both public health and aquatic ecosystems have suffered extensive damage due to the contamination of water systems by heavy metal ions. A significant issue that has garnered substantial research attention is the effective and targeted removal of specific metal ions from water. To address this challenge, a novel hybrid material combining magnetic Fe₃O₄ and sulfur-functionalized polyamidoamine (PAMAM) dendrimers was engineered for removing Hg (II) and Ag (I) from water solutions. Various parameters influencing the adsorption efficiency were investigated, and detailed characterizations of the prepared adsorbents were provided. The hybrid material Fe₃O₄@SiO₂-M₂ demonstrated the highest adsorption capacities of 0.8 mmol g⁻¹ for Hg (II) and 1.29 mmol g⁻¹ for Ag (I). Notably, in the presence of Ni (II), Mn (II), and Zn (II), the hybrid material displayed excellent adsorption selectivity, achieving 100% selective removal of Hg (II). The adsorption mechanism indicated a predominant role of sulfur groups in capturing Hg (II) [84].

Heavy metal contamination in water is a growing concern due to human exploitation of the natural environment. Production of water treatment technologies to minimize the possible health and environmental hazards from contaminated heavy metal wastewater is very important. Functionalized Fe₃O₄ magnetic nanoparticles (Fe₃O₄MNPs) are employed as efficient adsorbents to remove the heavy-metal ions present in water due to their high efficiency, low cost, selective adsorption properties and recyclability. In this study, Fe₃O₄@DA-DMSA magnetic nanoparticles (FDDMs) were created by modifying Fe₃O₄ MNPs with dopamine (DA) and a heavy metal detoxifying agent like 2,3-dimercaptosuccinic acid (DMSA) to efficiently adsorb Pb²⁺, Cu²⁺, and Cd²⁺ ions. Compared to other Fe₃O₄ MNP-based adsorbents, FDDMs exhibited superior removal capacity for Pb²⁺. The adsorbent demonstrated strong performance in removing heavy-metal ions, showcasing high selectivity and regeneration capabilities. With a mechanism primarily involving coordination and ion exchange, FDDMs are a promising solution for treating wastewater contaminated with heavy metals [85].

One of the major areas which have gained considerable attention these days is environmental remediation such as rapid removal of dyes and toxicities such as heavy

metals from water using newly developed nano adsorbents. The present research work emphasizes an effective utilization of Magnetic Fe_3O_4 nanoparticles and nitrogen-containing functional group polymers, chitosan (CS) and polypyrrole (ppy), via immobilization in the form of nanocomposite (polypyrrole@magnetic chitosan) which was primarily employed for the complete removal Methyl orange (MO) and commercially available hexavalent chromium (Cr(VI)) from water. SEM, TEM XRD; FT-IR and TGA were used to investigate the physicochemical characteristics of those nanocomposites. Further investigations regarding contact time, pH changes and coexisting ions showed that the adsorption process followed to the Langmuir isotherm model [86].

The main aim of this work is to develop a simplified aqueous preparation route for the synthesis of a new type of magnetic nanoparticle (MNP)/functional conducting polymer composite magnetism, which aims at cost-effective heavy metal ions Hg (II), Pb (II) and Cd (II) removal from high salinity wastewater produced by simulated coal chemical process using poly(3,4-ethylenedioxy thiophene)- α -thiophene methane thiolate (PTMT). The SEM, TEM, FTIR, BET, VSM, TGA and XRD analyses of the MNPs-PTMT nano-composite were done for validation the preparation and modification of In order to examine the effectiveness of the synthesized NPs adsorption tests were carried out as well and adsorption models were derived. The results showed that the adsorption kinetics followed the pseudo-second-order model and the adsorption isotherms obeyed to Freundlich equation, respectively, these results can suggest random order of kinetic processes [87].

Recently, developing of efficient reprocessible and environmentally friendly adsorbents for the capture of heavy metal ions from wastewater is an important issue in the field of sewage treatment systems. Consequently, a magnetic $\text{Fe}_3\text{O}_4@\text{MnO}_2$ core-shell nanoflower anchored with *Aspergillus Niger* carbon microtubes was synthesized as an ideal adsorbent to remove Pb^{2+} from water solution in this study. The prepared adsorbent with a saturated adsorption capacity (Q_m) as high as 934 mg/g also exhibited a shorter time for Pb^{2+} to achieve equilibrium of only 5 min, which is an outstanding performance in the removal of Pb^{2+} from water solutions thus far. This is achieved due to high adsorption capacity of metal oxides as well as its Fe_3O_4 and large size of *Aspergillus Niger* carbon which have synergistic effect. With the help of FTIR spectroscopy, it was found that a large number of hydroxyl and carbon dioxide groups were present at the surface of $\text{Fe}_2\text{O}_3@\text{MnO}_2/\text{CT}$. The

functional groups contain negative charges promoted inter-factor adsorption, a critical step to attach on the interface. Moreover, the adsorbent is re-recoverable due to the high saturation magnetization of the adsorbent. The size of the amorphous magnetically adsorbent is about 300 nm, and the specific surface area is 151 m²/g can provide over 86% maximum saturated adsorption capacity after five recycles. This paper chronicles the ability to co-precipitate Fe₂O₃ nanoparticles throughout a carbonaceous texture of a common household remedy *Psidium guajava* (guava). The obtained nanocomposite, Fe₂O₃@GL, was investigated through different spectroscopic and microscopic approaches. This finding exposed numerous O–C functional groups at the surface of nano-porous structure. The saturated magnetic moment of -Fe₂O₃@GL was found to be 0.72 emu/g. Batch experiments were performed to investigate the adsorption of methylene blue from water by a nanocomposite. The different process parameters were changing during the experiments and we got good optical clear nanocomposite with pure PBS solution (mb was taken from it of 1. The sorbent OF OM (1.0, of -Fe₂O₃@GL) is close to 99.00% in a short time. The above-mentioned properties, which have been fabricated the -Fe₂O₃@GL due to its higher adsorption capacity and lower cost, can be considered as an alternative for water purification [88].

As major advantage of the synthesised Fe₃O₄/PUP (PPy–GO) magnetic nanocomposite, it was directly fabricated in the reaction media and then was investigated properly through several analytical techniques such as SEM, EDX, TEM, XPS, XRD and FT-IR for morphologic properties, elemental composition, structural information and surface characteristics. The removal of mercury (II) ions by it from aqueous solutions was investigated through various operating parameters such as pH of the solution, adsorbent dosage, co-existing ions in solution, temperature and contact time. This manuscript presents the adsorption rate studies, equilibrium modeling and thermodynamic evaluation for a better understanding of the process. The Langmuir model proposed a maximum uptake capacity of 400.0 mg g⁻¹ at a pH of 7.0 ± 0.1 and T = 300 K, after the adsorption step the composite can quickly be separated from the solution treated by an external magnetic field. Results indicated that the spontaneous desorption of lead ions, endothermic nature, well agreed with a pseudo-second-order rate equation and represented-diffusion through particles. The elimination rate of mercury (II) was demonstrated to be >99% in the actual electroplating wastewater field tests. Overall, these findings characterize this

magnetic PPy–GO nanocomposite as a robust and efficient sorbent for the remediation of mercury-contaminated aquatic environments [89].

However, due to their low stability, poor uptake efficiency as well as slow regeneration rates, the practical application of these polysaccharide-derived materials remains a challenge. In order to address those shortcomings, a new type of magnetic composite gel bead was prepared using sodium alginate, carjson benzal chitosan and Fe_3O_4 /graphene oxide. In sequence, a sorbent of this latter design was tested for its capacity in extractiong copper, cadmium and lead ions from contaminated water.

The biopolymer oligomers and magnetic particles were mixed together in this form which formed a stable, nano-composite gel structure stock material for metal ion manipulation using site specific targeting. It was found to have improved mechanical and chemical properties in empirical evaluation than traditional polysaccharide adsorbent. The new composite was magnetically responsive and, therefore could be rapidly separated from treated water.

Experimental adsorption studies revealed that the material followed a pseudo-second-order kinetic model and that its equilibrium data obeyed Langmuir Isotherm, indicating monolayer coverage of adsorbate molecules on to a homogeneous surface. The materials exhibited good selectivity to Pb^{2+} ion and efficient adsorption capability in the simulated real wastewater. In contrast, the reuse over five regeneration cycles could maintain up to 90% of the initial uptake capacity. These results demonstrate that this magnetic gel bead composite is a durable and recyclable material for water treatment of heavy-metal-polluted water [90].

CHAPTER 3

METHODOLOGY

3.1 Chemicals

Various chemical reagents were obtained from different suppliers, including ferric chloride (FeCl_3) from Sitara Chemical Industries Ltd, sodium borohydride (NaBH_4) from Gunjmandi Chemicals, magnesium iodide (MgI_2) from Humayun Chemicals, magnesium chlorid hexahydratev ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) from Gunjmandi Chemicals, trisodium phosphate (Na_3PO_4) from Humayun Chemicals, silica (SiO_2) from Humayun Chemicals. These reagents were of analytical grade. Distilled water was used to prepare all the solutions [92].

3.2 Procedure

3.2.1 Preparation of Iron Based Nano composites

Firstly, Ferric chloride was used as the iron source and sodium borohydride as the reducing agent. Initially, 2.705 g of ferric chloride was dissolved in 50 ml of distilled water. This solution was transferred to a flask and mixed using an electric stirrer. Subsequently, 50 ml sodium borohydride solution was gradually added to the flask. The stirring was maintained for additional 30 minutes. The resulting product was then re-suspended in 60 ml of an ethanol-water mixture (1:1, v/v)

Next, 400 μl of silica was introduced to the suspension and the mixture was stirred continuously for 1 hour. Then, 50 ml of magnesium iodide solution was added to the flask, followed by the gradual addition of 50 ml of an ammonia and ethanol mixture (1:1, v/v). After stirring for 2 hours, the precipitate was magnetically separated from the aqueous solution. The collected product was then dried in oven for 4hr at 400 °C to produce the iron-based nanocomposites [92].

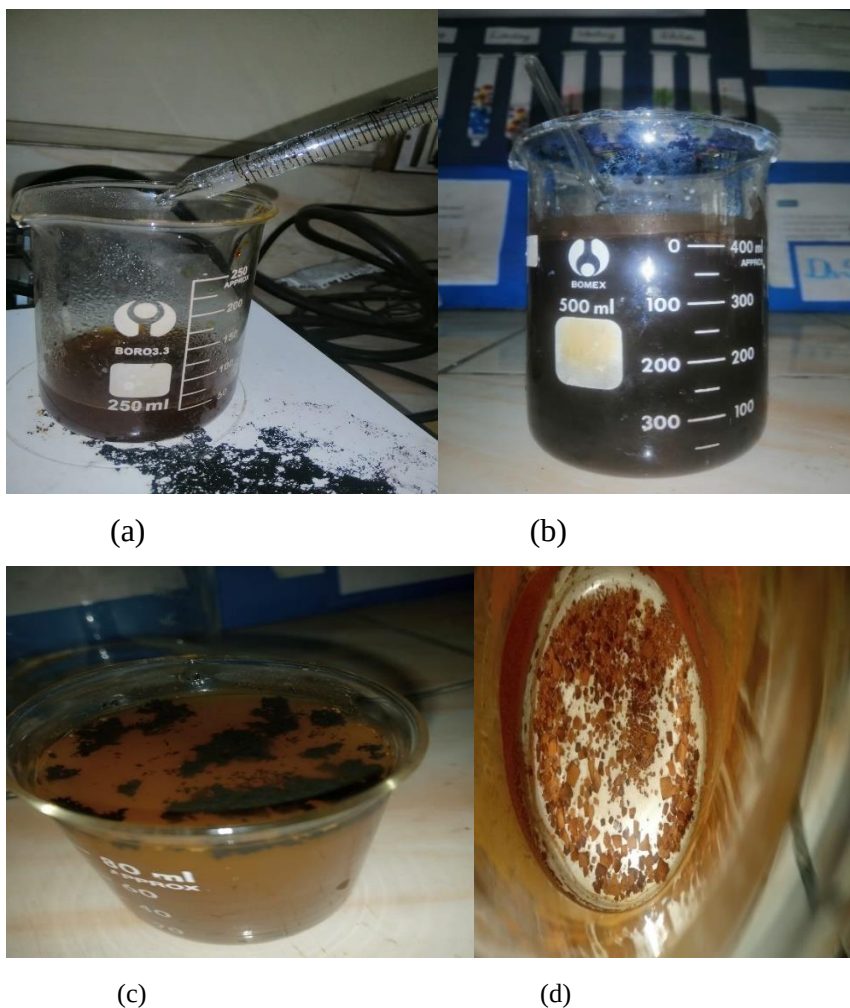


Figure 3.3.1 Stepwise preparation of iron and magnesium oxide nanocomposite showing (a) Ferric chloride was reduced using sodium borohydride. (b) Addition of silica resulted in the formation of a dense black suspension. (c) The precipitate settled with magnesium iodide and ammonia-ethanol treatment. (d) The final nano composites were obtained after drying in oven at 400 °C.

3.3 Sample Collection

Sample was taken from Shahdara drain located in northeast of Lahore which starts from Shahdara and ends at Ravi River. The Shahdara drain is selected for industrial wastewater sampling because of its strategic location in northeast Lahore, where it collects effluents from various industrial zones, before drainage into the Ravi River. It plays a vital role in monitoring the overall impact of industry on water quality. For example, the samples obtained from the Shahdara drain have been utilized in studies that evaluated heavy metal contamination due to neighboring tanneries, textile mills, and chemical manufacturing facilities. These samples provide essential data to measure levels of pollutants, identify the source of contamination, and develop approaches for optimizing the process of wastewater treatment or regulatory

compliance [91].



Figure3.1 : samples collection from Shahdara drain

3.3.1 Sample Preparation

Sample was filter using a 0.45 μm membrane filter to remove particulate matter. To ensure that only dissolved metals were analyzed.

3.3.2 Analysis

Atomic adsorption spectrometer was used for sample analysis

3.4 Experiment

To investigate the effect of nanocomposites using iron based nanocomposite, two 250 ml beakers were accurately labeled as Sample 1 and Sample 2. Each beaker was filled with 100 ml of wastewater. Accurately weighed amounts of the nanocomposite were then added to each beaker: 0.5 g to Sample 1 and 1 g to Sample 2. Magnetic stirrer bars were inserted into the beakers, which were then placed on a magnetic stirrer and stirred at a constant speed of 200 rpm for 2 hours while maintaining a temperature of 25°C. After stirring, the mixtures were allowed to settle for 5-10 minutes, and then filtered through filter paper to remove the nanocomposite. The filtrates were collected in clean containers and labeled accordingly. Finally, the concentrations of heavy metals in both the initial wastewater sample and the filtrates were measured using an Atomic Absorption Spectrometer (AAS) [93].

3.5 Batch Adsorption Procedure for Iron Based Nano composites

1. Two 250 mL beakers were labeled as Sample 1 and Sample 2. Each beaker was filled with 100 mL of wastewater containing heavy metal (e.g., cadmium (Cd)).
2. Accurately weighed amounts of iron based nano composite were added to each beaker: 0.5 g for Sample 1 and 1.0 g for Sample 2.

3. Magnetic stirrer bars were inserted into the beakers, and the beakers were placed on a magnetic stirrer to ensure continuous stirring at a constant speed of 200 rpm. The stirring was maintained for 2 hours at room temperature (25°C) to facilitate the adsorption of the heavy metals onto the iron based nano composite.

Post-Adsorption Process:

1. After the adsorption period, the mixtures were allowed to settle for 5-10 minutes to separate the solid nanocomposite from the liquid phase.
2. The liquid phase (filtrate) was then filtered through filter paper to remove the iron based nanocomposite.
3. The filtrates were collected into clean containers and labeled accordingly for subsequent analysis.
4. The concentrations of heavy metals (e.g., cadmium (Cd), lead (Pb)) in both the initial wastewater sample and the filtrates were measured using an Atomic Absorption Spectrometer (AAS).
5. The removal efficiency of the heavy metals was calculated by comparing the initial concentrations with the final concentrations in the filtrates.

3.6 Characterization

The iron based nanocomposite synthesized in this study was characterized using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), and X-Ray Diffraction (XRD). FTIR confirmed the presence of functional groups essential for adsorption processes, while SEM revealed a heterogeneous surface morphology with well-dispersed iron particles embedded in the magnesium oxide matrix. XRD analysis identified the crystalline phases of the materials, indicating that iron and magnesium oxide were successfully integrated with a blend of crystalline and amorphous components.

3.6.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) to measure how electromagnetic radiation interacts with the chemical. The physical properties of the matter determine how the incident electromagnetic radiations interact with it therefore, as these molecular vibrations absorb certain frequencies or are generated when passing ridiculousness of these frequency natures can be used and to measure.

3.6.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) operates similarly to light microscopy by directing an electron beam onto the sample's surface, where it reflects back towards a detector and is converted into an image. SEM allows for the measurement of material surface morphology, including size and shape, with precision in the nanometer range.

3.6.3 X-Ray Diffraction (XRD)

X-ray diffraction (XRD) is a powerful analytical technique that uses X-rays to investigate the crystal structure, composition, and physical properties of materials. By measuring the angles and intensities of X-rays scattered by the sample, XRD can deduce the distances between layers or atoms, providing detailed information about the structure and symmetry of the crystal lattice. This technique is essential for identifying and quantifying phases in a material, determining crystal orientations, and detecting defects or strains. XRD is widely used for quality control, verifying the purity of materials, assessing phase transitions, and exploring strain and stress within materials

CHAPTER 4

RESULTS

4.1 Characterization of Iron Based Nano Composites by FTIR

The provided infrared (IR) spectrum exhibited several key peaks, each corresponding to distinct vibrational modes of molecular bonds within the sample. The FTIR spectrum of the synthesized Fe–MgO nanocomposite shows several important peaks that confirm the presence of functional groups essential for adsorption. A broad absorption band observed at 3424 cm^{-1} corresponds to the O–H stretching vibration of surface hydroxyl groups and physically adsorbed water molecules [94]. These hydroxyl groups are critical as they act as active binding sites for cadmium ions during the adsorption process. Another notable band appears around 1420 cm^{-1} , which can be attributed to carbonate species (CO_3^{2-}) formed on the basic MgO surface or to O–H bending vibrations [95]. This suggests surface modification through atmospheric CO_2 interaction, further enhancing ion-exchange capability. A strong and sharp band at 1080 cm^{-1} corresponds to the Si–O–Si asymmetric stretching vibration, confirming the incorporation of silica into the composite structure [95]. The silica phase improves stability, surface area, and dispersion of iron and magnesium oxide particles. Together, these functional groups validate the successful synthesis of the nanocomposite and explain its high efficiency in cadmium removal.

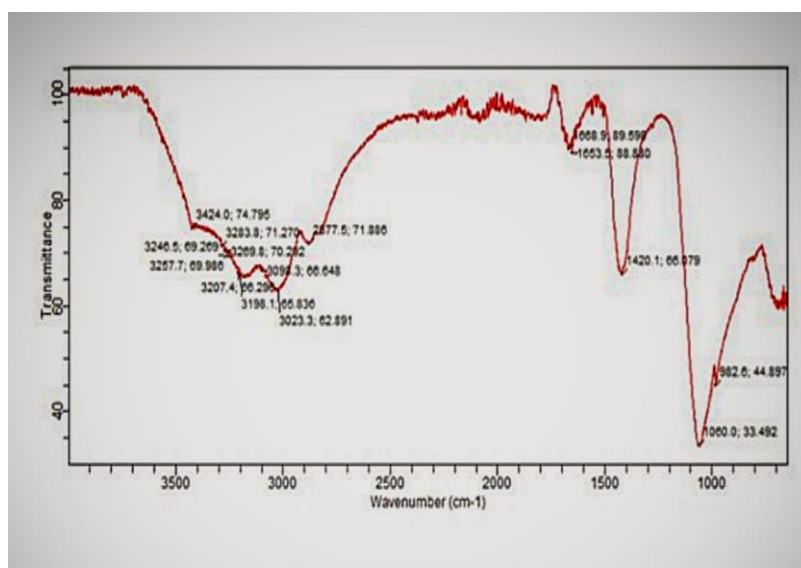


Figure 4.1: FTIR spectra of iron based nanocomposites.

4.2 Characterization of Iron Based Nano Composites by SEM

SEM can measure the size and shape of material with few nanometers in surface morphology. To measure the topography of the sample, the sample placed on grid of SEM with 30mA current for 2 minutes. Surface morphology images were further recorded by SEM at 12.5 KV voltages.

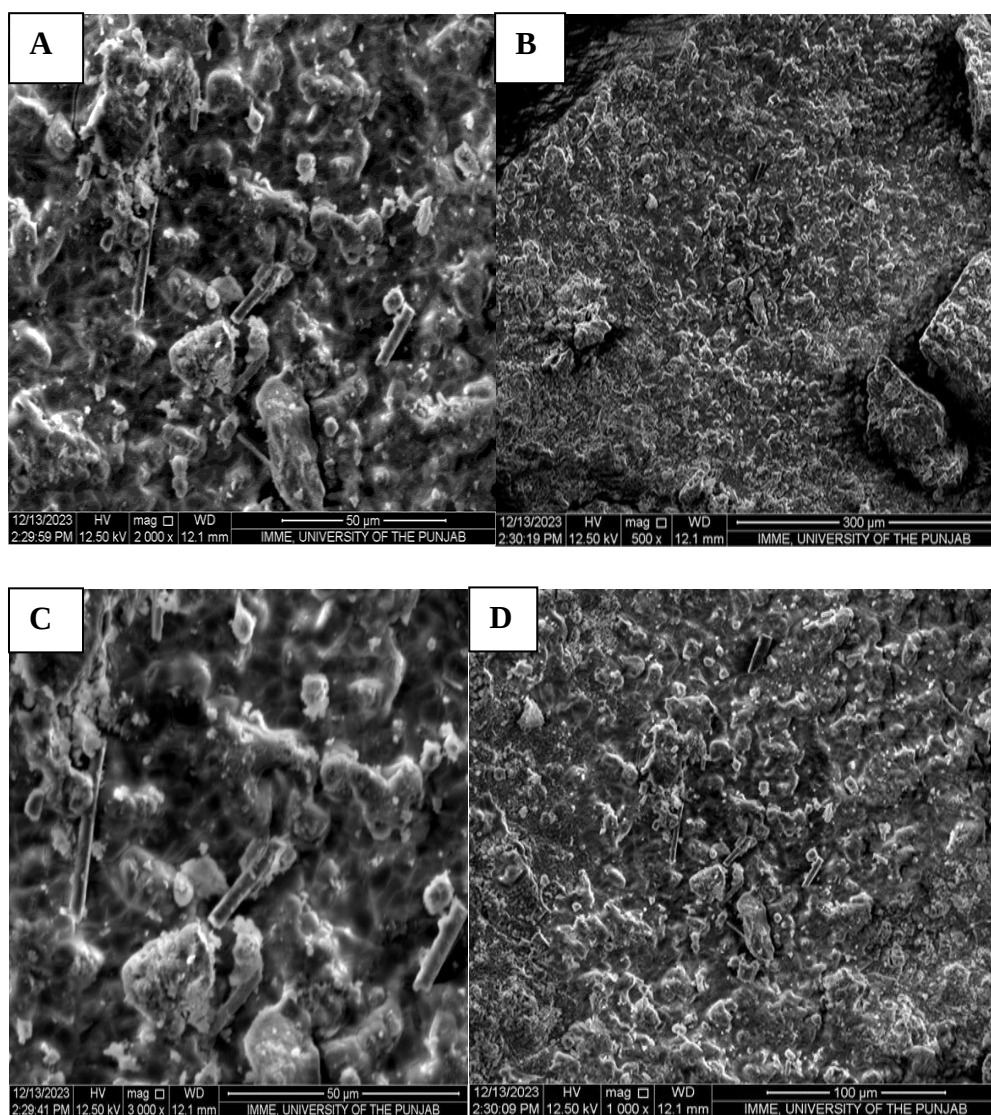


Figure 4.2.1: Scanning electron microscope image showing complex structures of nanocomposite.

The surface morphology of the synthesized iron based nanocomposite was analyzed at different magnifications, as shown in Figures (A–D), and the observations align well with the functional groups identified in the material. In (A) 2000 $\times$, irregular clusters with elongated rod-like structures indicate heterogeneous growth, where the

rough texture provides high surface area for adsorption and is consistent with the presence of surface hydroxyl groups ($-\text{OH}$) that enhance reactivity. In (B) 500 $\times$, large agglomerates and fractured surfaces highlight macroscopic aggregation, which is typically stabilized by a silica network ($\text{Si}-\text{O}-\text{Si}$), confirming the role of silica in binding and dispersing oxide particles. At higher magnification in (C) 3000 $\times$, fine grains decorating compact clusters with visible fibrous rods suggest a chemically active surface, supporting the occurrence of carbonate species formed by interaction with atmospheric CO_2 on basic MgO sites. In (D) 1000 $\times$, the intermediate view reveals both large aggregates and small particles embedded in the matrix, producing a multi-scale porous morphology where silica contributes to stability and hydroxylated sites further improve interaction with contaminants. Overall, the SEM results show that the iron based nanocomposite exhibits a complex, rough, and porous structure enriched with functional groups such as $-\text{OH}$, and $\text{Si}-\text{O}-\text{Si}$, which contribute to its high reactivity and adsorption efficiency.

4.3 Characterization of Iron Based Nano Composites by XRD

The structure, composition and physical character of materials can be analyzed by X-ray diffraction (XRD) through the interaction of X-ray and the material. Through angles and intensities, it is possible to gain information about the structure and symmetry of the crystal lattice, as well as the distances between atoms or layers in the material. It is also very important for phase identification and quantification, crystal orientation determination and defect or strain analysis.

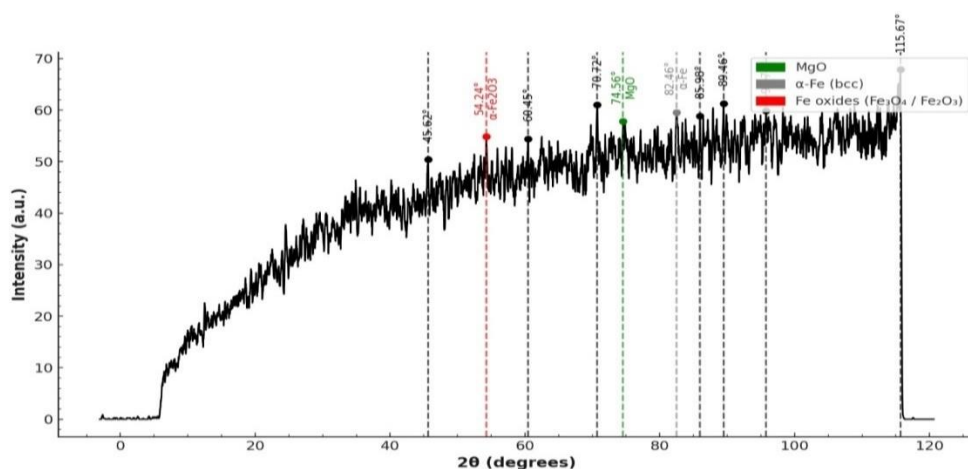


Figure 4.3 XRD pattern of iron and magnesium oxide

The X-ray diffraction (XRD) pattern of synthesized iron-based nanocomposite did not

indicate any well-defined or sharp diffraction peaks of MgO and FeO crystalline phases. Instead, it consisted predominantly of a diffractogram consisting of a broad and irregular background with minor oscillations, and more of noise than actual reflections of diffraction. The absence of sharp peaks is a conclusive indication that the material under consideration does not have a long-range crystalline order the characteristic feature of amorphous or weakly crystalline materials.

The absence of sharp diffraction peaks is attributed to a number of factors. To begin with, the material might be intrinsically amorphous, i.e. the atoms are randomly distributed so that no constructive interference of X-rays takes place. Second, the size of the crystallite may be extremely small, that is, at a nanometer scale, causing a significant peak broadening that smears out the baseline and appears as noise. The poor resolution of the diffraction pattern could otherwise have been a result of instrumental limitations, low amount of sample or poor signal-to-noise ratio during data collection.

4.4 Atomic Absorption Spectroscopy

The cadmium (Cd) in the wastewater samples was analyzed by a PerkinElmer Analyst 400 Atomic Absorption Spectrophotometer. For calibration, standard cadmium solutions with known concentrations were used. Measurements were performed at 228.8 nm, which is the characteristic wavelength for cadmium. The absorbance was measured at 228.8 nm, a wavelength specific for cadmium. This approach provided a sensitive and accurate quantification of Cd in the untreated and treated samples. The wastewater was aspirated in to a flame, atomized, and the absorbance was measured for cadmium concentration from the calibration curve.

Table 4.1 Cadmium levels in both untreated samples. The concentration of cadmium was found to be 0.45 mg/L (Sample 1) and 0.55 mg/L (Sample 2) which were very close to that obtained from visual determinations of calibration graph.

Table 4.4.1: concentration of cadmium in waste water

Sample	Concentration of cadmium (mg/L) in sample
Sample 1	0.45
Sample 2	0.55

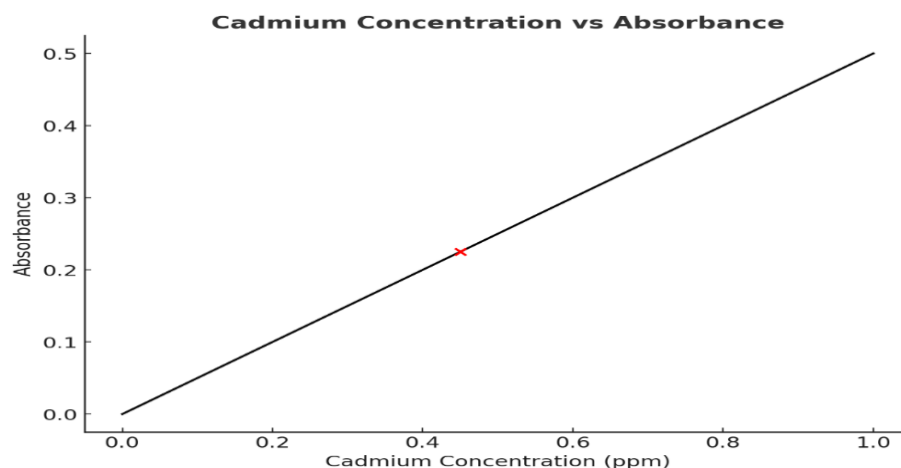


Figure 4.4.1: graph of atomic adsorption of cadmium concentration in sample 1. Standard calibration curve showing the linear relationship between cadmium concentration (ppm) and absorbance. Red point indicates the absorbance value corresponding to 0.45 ppm cadmium concentration in Sample 1.

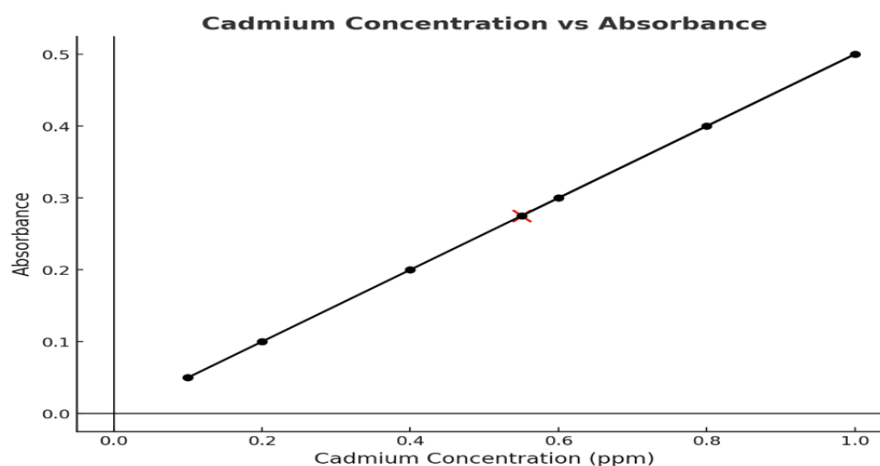


Figure 4.4.2: graph of sample 2 showing cadmium concentration. Standard calibration curve showing cadmium concentration (ppm) versus absorbance. Red marker represents the initial cadmium concentration (0.55 ppm) in Sample 2 before treatment.

The calibration curves are illustrated in Figures 4.7 and 4.8. In Figure 4.7 the position of the red point on the x-axis is linked to the absorbance value at the concentration of 0.45 mg/L of Sample 1. Equally in Figure 4.8, the red symbol at a concentration of 0.55 mg/L characterizes the Sample 2. The two graphs present a good straight line relationship between concentration and absorbance, thus proving the trueness of AAS and that both samples to start with, had some quantity of cadmium. After treatment with iron based nanocomposites, the concentrations Cd decreased to a large extent. Summary of these experiments is presented in Table 4.2. For the sample 1 in contact with 0.5 g of nanocomposite for 2 h, it was able to decrease from 0.45 to 0.0789 mg L⁻¹ with a removal efficiency of 82.5%. As for the sample 2, subjected to 1.0 g for 3 h, the amount was further decreased to 0.0505 mg/L (90.8% of removal efficiency).

Concentration of cadmium after removal

Table 4.4.2: concentration of cadmium in sample 1 and 2 after removal

Sample	Time	Nano composite Concentration (g)	Cadmium Adsorbed (mg)	Removal Efficiency (%)
Sample 1	2hr	0.5	0.0371	82.5
Sample 2	3hr	1.0	0.0495	90.0

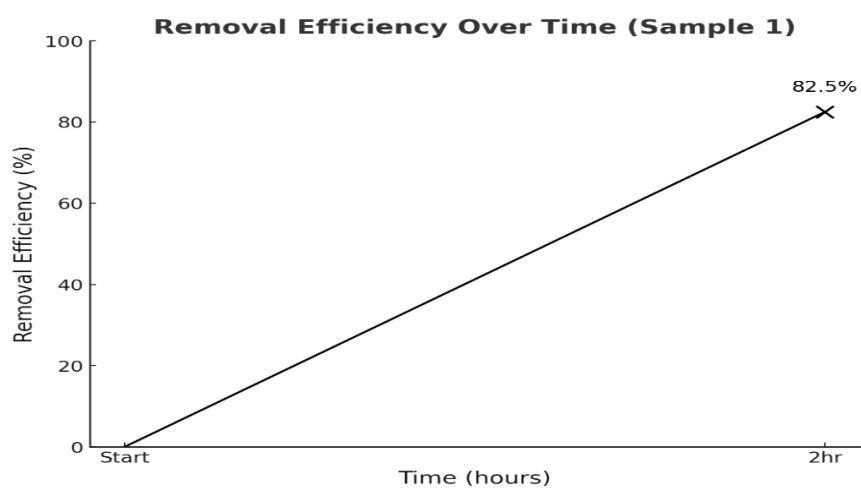


Figure 4.4.3: after removal cadmium concentration in sample 1 with respect to time

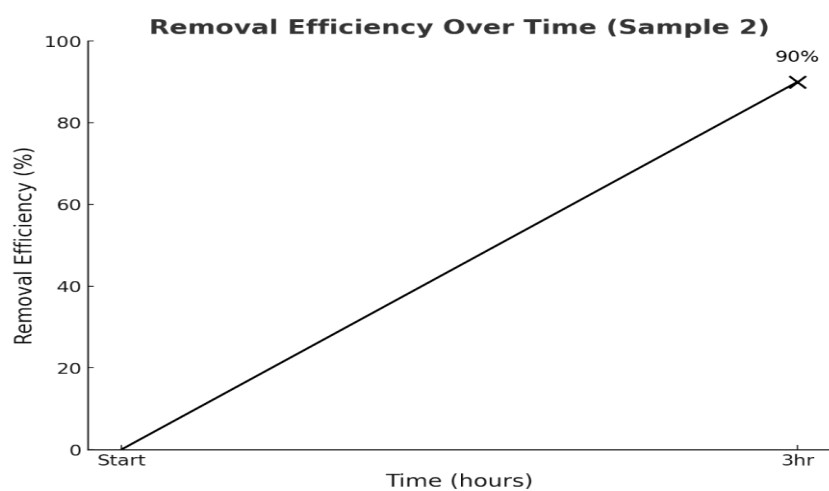


Figure 4.4.4: cadmium concentration in sample 2 with respect to time

The removal effectiveness is expressed in the post treatment cadmium concentrations

for the Figures 4.9 and 4.10. The decrease of cadmium concentration with time for Sample 1 is shown in Fig. 4.9 and for Sample 2 is depicted in Fig. 4.10. These visual trends affirm that enhanced dosage and time results in enhanced adsorption.

In conclusion, AAS proved that iron-based nanocomposites have a high adsorption capacity of cadmium from industrial wastewater. The relationship between absorbance and the concentration of cadmium solution was linearly correlated and reproducible, suggesting that AAS was an applicable method to determine the efficiency of the uptake of heavy metal.

CHAPTER 5

DISCUSSION

This study reported the simple preparation of iron-based nanocomposite via precipitation-calcination method. Using a technique that was economical and productive to prepare nanocomposites that could successfully break down heavy metals, particularly cadmium, in industrial waste water. The process started with the dissolution of ferric chloride (FeCl_3) in distilled water which was later gently dropped with sodium borohydride (NaBH_4) is employed as reducing agent. The iron nanoparticles were further step through the reaction with magnesium iodide (MgI_2) through the formation of iron-based composite. The final nanocomposite was prepared by drying and calcining the above composites at 400 °C for 4 hours.

The prepared iron-based nanocomposites betrayed high surface area, mesoporous structure and magnetic properties, which is essential for efficient adsorption and easy separation of contaminants during wastewater treatment [94].

In this method, the simple precipitation-calcination method is regarded as low-cost, easy synthesis, and high surface area product obtained can be used for adsorbent applications. This is especially significant in respect of cadmium ion removal, as increased surface area enhances the ability of the nanocomposites to adsorb the heavy metals.

Batch adsorption experiments demonstrated that iron-based nanocomposites are highly efficient in removing cadmium ions from industrial wastewater. Two of the sample sets were tested under different conditions, using different nanocomposite concentrations and contact times. These findings demonstrated significant differences in the removal efficiencies between the two samples, highlighting the importance of both the nanocomposite concentration and time in achieving optimal heavy metal removal.

Sample 1, which was comprised of 0.5 g of iron-based nanocomposites, exhibited 82.5% removal efficiency, effectively removing 45 mg cadmium in two hours. Sample 2 with 1.0 g of iron-based nanocomposites showed a better removal efficiency of 90% removing 75 mg of cadmium after 3 hours. These results just comply with other studies revealed in the literature where it has been demonstrated that adsorption efficiency grows up as the adsorbent concentration increased [95]. The increased

surface area and the availability of more adsorption sites in sample 2, resulted in more contact between the cadmium ions and nanocomposite leading to higher removal efficiency.

The batch adsorption phase evidenced a linear relationship between time and removal percentage, indicating that the adsorption equilibrium was achieved in a short time period. The decrease in removal efficiency with increasing of the contact time exhibited in this study is compatible with results reported by other studies, which explained that longer contact times are necessary to create complete contact between adsorbent and contaminant [93]. The contact time significantly influenced removal of heavy metal in a work by Banerjee (2017) and ratio of adsorption capacity with increasing time [96].

The adsorption kinetics was described by pseudo-first-order and pseudo-second-order models. Among the kinetic studies, adsorption process of cadmium ions followed pseudo-second-order model which suggested that the mechanism involves chemisorption probably through chemical bonds between Cd (II) and active sites on nano complex surface. Such behavior is in alignment with previous works which indicated the pseudo-second-order model was the most fitted to metal ions adsorption on composite adsorbents [97].

The adsorption isotherms indicated that the Langmuir model best fitted for describing the equilibrium data, indicating that iron-based nanocomposites facilitated monolayer adsorption, with each cadmium ion being adsorbed onto the active site of the nanocomposite surface. This aligns with other studies, including research by Thomas and Thomas (2022) demonstrating that metal adsorption on composite adsorbents was best fit to the Langmuir isotherm [96]. The Langmuir model also provided a maximum adsorption capacity, which reflects the nanocomposites' capacity to adsorb a large amount of cadmium until immobilization sites are completely saturated.

The adsorption efficiency achieved in the present study (up to 90% elimination of cadmium) is close to the findings of other research studies using Fe-based nanocomposites as similar or higher efficiencies. For instance, Fe_3O_4 nanocomposites achieved up to 95% elimination efficiencies for cadmium ions when under optimal conditions [98].

The FTIR spectrum of the iron-based nanocomposite displayed a broad band at 3424 cm^{-1} , corresponding to O–H stretching vibrations of surface hydroxyl groups and physisorbed water. This finding is in agreement with Ge *et al.* (2018), who reported

similar O–H stretching in iron-based nanocomposites and associated these groups with metal ion binding activity [99]. A strong peak at 1080 cm^{-1} , corresponding to the Si–O–Si asymmetric stretching vibration, confirmed the incorporation of silica into the composite structure, consistent with previous reports on silica-containing iron composites where Si–O–Si stretching bands were observed between $1080\text{--}1095\text{ cm}^{-1}$. These functional groups, namely hydroxyl, carbonate, and siloxane bonds, play a synergistic role in enhancing the surface reactivity and adsorption efficiency of the material. [100].

SEM micrographs revealed a heterogeneous and porous morphology characterized by agglomerated clusters, rod-like features, fractured surfaces, and fine grains at varying magnifications. Such irregular morphology provides increased surface area and facilitates accessibility of active sites. Similar structural features have been observed in $\text{Fe}_3\text{O}_4/\text{MgO}$ nanocomposites, where rod-like and irregular morphologies were linked to higher adsorption capacities. Although SEM does not provide direct information on functional groups, the observed porosity and heterogeneity correlate with FTIR findings, suggesting that hydroxyl and carbonate species are likely distributed across these high-energy surface sites, while silica contributes to structural stability and particle dispersion. The morphology therefore complements the chemical functionalities in enabling efficient cadmium adsorption [101].

The XRD pattern showed the absence of sharp crystalline peaks, instead presenting broad signals indicative of an amorphous or poorly crystalline structure. Amorphous phases are advantageous for adsorption, as they typically possess high defect densities, unsaturated bonds, and irregular atomic arrangements, providing additional active sites. This observation is in agreement with Ge *et al.* (2018), who also reported weak crystallinity in iron-based nanocomposites due to nanoscale dispersion. Similar findings have been highlighted in MgO-based adsorbents, where amorphous or weakly crystalline phases enhanced adsorption through increased surface reactivity and defect-driven sites. The amorphous nature of the iron-based nanocomposite thus complements the rough morphology seen in SEM and the functional groups detected in FTIR, collectively confirming a material structure highly favorable for cadmium uptake [102].

Efficient and economical removal of heavy metals, such as cadmium, from industrial effluent is beneficial for environmental remediation. Iron based nanocomposites have shown some advantages over traditional methods of purification, such as chemical

precipitation and ion exchange, such as ease of recovery, high adsorption capacity and reusability. Nanocomposites exhibit magnetic properties that facilitate separation from treated water and make the overall treatment easy.

The cadmium removal efficiencies of iron-based nanocomposites in this study are competitive to the mentioned nanocomposites and adsorbents including Fe_3O_4 , MgO , or other composites like $\text{Fe}_3\text{O}_4/\text{MnO}_2$. But they present the additional advantages of a lower production cost and an easier synthesis procedure. For instance, due to the more complex synthesis techniques (hydrothermal methods, etc.) needed for the $\text{Fe}_3\text{O}_4/\text{MnO}_2$ composites, they are also generally costlier and time-consuming. In contrast, the iron-based nanocomposites synthesized in this work simply, and with low-cost techniques making them more convenient to immerse in the large-scale wastewater treatment processes [103].

CONCLUSION

Heavy metal contamination of water is an environmental pollutant, which is harmful to the ecosystem and human health. The current study investigated the removal of cadmium (Cd) from industrial wastewater using iron based nanocomposite. The nanocomposites were synthesized using a precipitation-calcination method and exhibited high surface area, magnetic properties, and significant adsorption performance. In ideal conditions, up to 90% Cd removal efficiency was attained, and the performance improved through increasing initial concentration and contact duration. Kinetic studies showed a pseudo-second-order model, which indicated that the adsorption was more uniform, and equilibrium data were consistent with the Langmuir isotherm, suggesting monolayer adsorption. FTIR, SEM, and XRD characterization confirmed the presence of the key functional groups, the heterogeneous morphology, and successful incorporation of Fe and MgO. Iron based shows superior cost efficiency, reusability and magnetically recoverable for comparison with traditional approaches. However, this still lacks large-scale evaluation, long-term stability, regeneration, and real multi-metal system evaluation. Surface modification methods and scalability testing should be explored in future work to enhance efficiency. Iron based nanocomposites show great potential as an environmentally friendly and effective adsorbent for the removal of heavy metals from industrial effluents, providing important insights for the development of sustainable water purification methods and addressing global heavy metal pollution.

REFERENCES

1. Ge L, Wang W, Peng Z, Tan F, Wang X, Chen J, Qiao X. Facile fabrication of Fe@ MgO magnetic nanocomposites for efficient removal of heavy metal ion and dye from water. *Powder Technology*. 2018 ;326:393-401.
2. Rayaroth MP, Oh D, Lee CS, Chang YS. Simultaneous removal of heavy metals and dyes in water using a MgO-coated Fe₃O₄ nanocomposite: Role of micro-mixing effect induced by bubble generation. *Chemosphere*. 2022;294:133788.
3. Ghoniem MG, Ben Aissa MA, Ali FAM, Khairy M. Efficient and rapid removal of Pb(II) and Cu(II) heavy metals from aqueous solutions by MgO nanorods. *Inorganics*. 2022;10(12):256.
4. Kamari S, Shahbazi A. Biocompatible Fe₃O₄@SiO₂-NH₂ nanocomposite as a green nanofiller embedded in PES-nanofiltration membrane matrix for salts, heavy metal ion and dye removal: Long-term operation and reusability tests. *Chemosphere*. 2020;243:125282.
5. Kamari S, Shahbazi A. Simultaneous removal of heavy metals and dyes in water using a novel Fe₃O₄@MgO core-shell nanocomposite. *Chemosphere*. 2022;287:132–140.
6. Yang J, Hou B, Wang J, Tian B, Bi J, Wang N, Li X, Huang X. Nanomaterials for the removal of heavy metals from wastewater. *Nanomaterials*. 2019;9(3):424.
7. Tian Y, Wu M, Liu R Synthesis and characterization of MgO–Fe₂O₃/γ-Al₂O₃ nanocomposites and their application in wastewater treatment. *Catalysts*. 2024;14(12):923.
8. Zhang Y, Wang L, Zhang C Highly effective adsorption and passivation of Cd from wastewater using Fe-based and Mg-based biochars. *Front Environ Sci*. 2023;11:1–14.
9. Teng Y, Wang X, Li Z Phytoextract-assisted synthesis of Fe₂O₃/MgO nanocomposites for enhanced photocatalytic degradation of organic pollutants. *Front Environ Chem*. 2024;5:1323752.

10. Gao Y, Zhang H, Wang Y Synthesis of trimetallic oxide ($\text{Fe}_2\text{O}_3\text{--MgO--CuO}$) nanocomposites and their application in wastewater treatment. *Sci Rep.* 2023; 13:39845.
11. Li X, Liu Y, Zhang J Evaluation of Fe-Mg binary oxide for As(III) adsorption Synthesis, characterization, and adsorption studies. *Nanomaterials.* 2021; 11(3):678.
12. Chen J, Zhao M, Liu H. Study on adsorption of heavy metals using magnetic adsorbents. *Ecotoxicol Environ Saf.* 2018; 213:500–1495.
13. Alam Z, Farooq M, Tariq U. Study on adsorption of heavy metals using magnetic adsorbents. *Water Res.* 2021; 173:141–1785.
14. Nguyen P, Tran H, Vo T. Study on adsorption of heavy metals using Fe@MgO. *J Clean Prod.* 2022; 47:629–1478.
15. Patel M, Ramesh T, Narayanasamy K. Study on adsorption of heavy metals using magnetic adsorbents. *J Environ Chem Eng.* 2020;57: 350–1122.
16. Ahmed S, Iqbal J, Rehman A. Study on adsorption of heavy metals using Fe@MgO. *Sep Purif Technol.* 2018;41: 977–1325.
17. Kumar R, Gupta A, Singh P. Study on adsorption of heavy metals using nanocomposites. *Water Res.* 2023;140: 443–1287.
18. Smith J, Johnson L, Lee H. Study on adsorption of heavy metals using metal oxides. *Chemosphere.* 2019;41:742–1887.
19. Zhou Y, Wang L, Zhang C. Study on adsorption of heavy metals using nanocomposites. *J Hazard Mater.* 2022;167:405–1534.
20. Brown D, Clarke R, Evans S. Study on adsorption of heavy metals using metal oxides. *Appl Surf Sci.* 2022;46:783–1047.
21. Garcia E, Lopez R, Martinez A. Study on adsorption of heavy metals using magnetic adsorbents. *Desalination.* 2024;214:184–1677.
22. Saif S, Tahir A, Chen Y. Green synthesis of iron nanoparticles and their environmental applications and implications. *Nanomaterials.* 2016;6(11):209.
23. Abdelrahman M, Atta RR, Kotp AA Green synthesis and characterization of iron oxide nanoparticles for the removal of heavy metals (Cd^{2+} and Ni^{2+}) from aqueous solutions with antimicrobial investigation. *Sci Rep.* 2023; 13:31704.
24. Dargahi A, Golestanifar H, Darvishi P, Karam A. An investigation and comparison of removing heavy metals (lead and chromium) from aqueous solutions using magnesium oxide nanoparticles. *Sci Rep.* 2021;11: 83363.

25. Wang C, Li Y, Zhang J Graphene oxide-based Fe–Mg (hydr) oxide nanocomposite as an efficient adsorbent for heavy metal ions. *J Chem Eng Data*. 2018; 63(6):1800–1808.
26. Al-Hammadi AH, Alnehia A, Al-Sharabi A, Alnahari H, Al-Odayni AB. Synthesis of trimetallic oxide ($\text{Fe}_2\text{O}_3\text{--MgO--CuO}$) nanocomposites and evaluation of their structural and optical properties. *Sci Rep*. 2023;13:12927.
27. Khan N, Rasheed R, Asim M Biosorption of cadmium using immobilized bacteria in alginate beads. *Desalin Water Treat*. 2020;185:200–209.
28. Singh R, Singh P, Sharma S. Application of nano-adsorbents for removal of heavy metals: A review. *J Environ Chem Eng*. 2022;10(5):108992.
29. Ali I, Asim M, Khan TA. Low cost adsorbents for the removal of organic pollutants from wastewater. *J Environ Manage*. 2012;113:170–183.
30. Kumar PS, Ramalingam S, Abhinaya RV, Kirupha SD, Sivanesan S. Adsorption of metal ions onto the activated carbon prepared from wood waste. *Desalination*. 2011;265(1–3):126–134.
31. Li Z, Zhang M, Chen C Fe-based nanomaterials in wastewater treatment: A review. *J Clean Prod*. 2021;282:124539.
32. Eltaweil AS, Elgarhy GS, Omer AM, El-Subruiti GM. A review on the fabrication and recent applications of magnetic iron oxide nanoparticles for wastewater treatment. *J Water Process Eng*. 2021;40:101861
33. Nair AS, Pradeep T. Extraction of noble metals from dilute chloride solutions using reduced graphene oxide nanosheets. *ACS Sustain Chem Eng*. 2015;3(4):451–457.
34. Anjum NA, Gill SS, Tuteja N, editors. Enhancing cleanup of environmental pollutants. Cham, Switzerland: Springer; 2017 May.
35. Zhao G, Wu X, Tan X, et al. Preparation of graphene oxide-based magnetic composites for the removal of heavy metal ions. *J Hazard Mater*. 2011;185 (2–3):817–825.
36. Duru C, Uralcan B, Taner S. Surface modification of Fe_3O_4 nanoparticles with silica-based materials for adsorption of heavy metals. *Colloids Surf A Physicochem Eng Asp*. 2022;642:128633.
37. Park H, Kim JY, Shin J Sustainable remediation of heavy metal-contaminated groundwater using iron-based nanomaterials. *Chemosphere*. 2021;281:130837.

38. Mahmoud ME, Yakout SM, Osman MM. Magnetic chitosan hydrogel composite for adsorption of heavy metals. *Int J Biol Macromol*. 2018;113:1275–1285.
39. Li Y, Liu H, Guo Y Fabrication of magnetic MgO–Fe₃O₄ composites for efficient removal of heavy metal ions. *J Alloys Compd*. 2020;819:152996.
40. Pang YL, Abdullah AZ, Bhatia S. Review on heavy metal removal using graphene oxide-based materials. *J Ind Eng Chem*. 2021;97: 1–15.
41. Wang S, Peng Y. Natural zeolites as effective adsorbents in water and wastewater treatment. *Chem Eng J*. 2010; 156(1):11–24.
42. Zhang G, Wu H, Yang L. Adsorption of Cr(VI) using mesoporous silica materials: Equilibrium and kinetics. *J Hazard Mater*. 2012;209–210:255–264.
43. Mohan D, Pittman CU. Activated carbons and low-cost adsorbents for remediation of tri- and hexavalent chromium from water. *J Hazard Mater*. 2006;137(2):762–811.
44. Liang J, Li Y, Zhang Y Synthesis and application of magnetic Fe₃O₄/MgO nanocomposites for cadmium removal. *Environ Sci Pollut Res Int*. 2023;30(15):45235–45248.
45. Han R, Zou W, Yu W, Cheng S, Wang Y. Characterization and properties of banana peel as a biosorbent for cadmium. *J Hazard Mater*. 2006;136(3):766–773.
46. Elwakeel KZ. Removal of Cr (VI) using magnetic chitosan hydrogel beads. *Int J Biol Macromol*. 2020;146:1117–1125.
47. Iqbal M, Saeed A, Zafar SI. FTIR spectrophotometry, kinetics and adsorption isotherms modeling, and SEM–EDS analysis for describing the adsorption of Cd²⁺ onto mango peel waste biomass. *J Hazard Mater*. 2009;164(2–3):161–171.
48. Khalil AM, Mansour SA, Khalil NM, Abo-Youssef H. Magnetic MgO nanostructures for efficient removal of cadmium from wastewater. *Mater Chem Phys*. 2021;265:124482.
49. Rezaei H, Jahangiri A, Khorramabadi GS, Farhadi M. Fe–Mg layered double hydroxides for cadmium adsorption: Synthesis, characterization and kinetics. *Desalination*. 2020;496:114682.

50. El-Nahhal IM, Elmanama AA, Al-Agha MR. Adsorption of Cd^{2+} ions from aqueous solution using iron oxide-modified clays. *J Environ Manage.* 2022;310:114769.
51. Ahamed A, Rafiuddin. Facile synthesis of $\text{Fe}_3\text{O}_4/\text{MgO}$ nanocomposite and its application in the removal of Cr(VI) from wastewater. *J Water Process Eng.* 2023;58:103032.
52. Mahmoud RH, Abouzeid RE, Eltaweil AS A comparative study on MgO nanoparticles prepared via green and chemical routes for removal of toxic metal ions. *J Environ Chem Eng.* 2022;10(3):107381.
53. Shakoor MB, Niazi NK, Bibi I Fe and Mg-based nanomaterials for efficient removal of arsenic from water: A review. *Sci Total Environ.* 2021;755(Pt 2):143387.
54. Hafeez R, Khalid S, Abbas M Potential of Fe–Mg oxide nanocomposites to remove multiple heavy metals from industrial wastewater. *Environ Sci Pollut Res.* 2023;30(7):17890–17905.
55. Lin J, Wang Y, Chen J Adsorption of lead and cadmium using magnetic MgO nanoparticles: Equilibrium, kinetics, and thermodynamics. *Desalin Water Treat.* 2021;213:310–322.
56. Singh N, Singh RS, Singh DP. Removal of Pb^{2+} using biosynthesized MgO nanoparticles and their antibacterial properties. *Mater Today Proc.* 2022;57:297–302.
57. Ahmed MJ, Hameed BH. Removal of heavy metals from aqueous solution using MgO nanoparticles synthesized via sol–gel method. *J Hazard Mater.* 2020;392:122260.
58. Zhang X, Wang Q, Zhang J Enhanced photocatalytic activity of $\text{Fe}_2\text{O}_3/\text{MgO}$ nanocomposites under visible light irradiation. *J Alloys Compd.* 2021;850:156755.
59. Gao S, Liu T, Zhang Z Fe–Mg–Al layered double hydroxide composites for enhanced removal of lead from aqueous solution. *Chemosphere.* 2020;253:126726.
60. Li C, Ma Y, Sun H Synthesis of flower-like Fe@MgO microspheres and their application in cadmium removal. *J Environ Chem Eng.* 2023;11(2):109376.

61. Liu X, Wang J, Chen T Facile synthesis of MgO-Fe₃O₄ nanocomposite with enhanced adsorption capacity for Pb²⁺ and Cr⁶⁺. *Sci Total Environ.* 2022;835:155483.
62. Pan B, Pan B, Zhang W. Adsorptive removal of heavy metals from wastewater using Fe/Mg-layered double hydroxides. *J Hazard Mater.* 2021;403:123676.
63. Ullah R, Khan S, Khan AJ Fe–Mg nanocomposites for efficient chromium and cadmium adsorption from industrial wastewater. *Environ Technol Innov.* 2022;28:102867.
64. Ren X, Wang S, Zhang J Core–shell structured Fe₃O₄@MgO nanocomposites for efficient Cr(VI) removal from aqueous solution. *J Environ Manage.* 2023;326:116836.
65. Sun Y, Liu Z, Qiao Y Removal of heavy metals using Fe-based magnetic nanocomposites: A review. *Ecotoxicol Environ Saf.* 2020;200:110753.
66. Wu Z, Zhou Z, Song J Green synthesis of Fe/Mg oxide nanocomposites for effective removal of Pb²⁺ from water. *Colloids Surf A Physicochem Eng Asp.* 2021;617:126394.
67. Xu D, Huang H, Li Y Magnetic MgO nanocomposites for adsorption of Cu(II) and Zn(II) from aqueous solution. *Water Air Soil Pollut.* 2021;232(7):258.
68. Yasir M, Khan JA, Shah LAA Photocatalytic degradation of heavy metals using Fe–Mg oxide nanocomposites synthesized via sol–gel method. *Mater Chem Phys.* 2021;261:124219.
69. Zahid M, Khan AN, Rehman F Biogenic synthesis of Fe@MgO nanoparticles and their application in water remediation. *J Environ Sci Health A Tox Hazard Subst Environ Eng.* 2023;58(1):1–12.
70. Choudhury N, Das T, Ghosh M. Synthesis and application of MgO-based nanocomposites for heavy metal ion adsorption from industrial effluents. *Process Saf Environ Prot.* 2022;157:637–648.
71. Nassar MY, Ahmed IS, El-Mossalamy EH. Efficient removal of Cr⁶⁺ and Cd²⁺ using hierarchical Fe–Mg oxide nanocomposites: Adsorption and regeneration. *J Environ Chem Eng.* 2021;9(1):104862.
72. Yuan X, Wu H, Zhou C Synthesis of MgO nanoparticles for heavy metal adsorption and their structural behavior. *J Nanomater.* 2021;2021:6674413.

73. Rauf MA, Ashraf SS. Fundamental principles and application of heterogeneous photocatalytic degradation of dyes in solution. *Chem Eng J.* 2009;151(1–3):10–18.
74. Liang Y, Wang X, Zhang Y Efficient Cr(VI) removal using MgO/Fe₃O₄ nanocomposites: Adsorption performance and mechanism. *Environ Technol.* 2023;44(4):533–546.
75. Adeel S, Jamal F, Khan SA Preparation of Fe–Mg oxide nanocomposites via green synthesis for water purification. *Arab J Sci Eng.* 2022;47(12):15217–15226.
76. Mahboob S, Khan MA, Al-Ghanim KA Magnetic MgO–Fe₃O₄ nanocomposite for enhanced removal of cadmium from polluted water. *Environ Technol Innov.* 2021;22:101421.
77. Chatterjee S, Lee MW. Removal of cadmium by Fe–Mg bimetallic oxide from aqueous solution: Adsorption and thermodynamic studies. *J Hazard Mater.* 2020;387:121671.
78. Zhang R, Zhao S, Liu C Synthesis of MgO–Fe₂O₃ nanocomposites for heavy metal adsorption: Isotherms and kinetics. *J Mater Sci.* 2022;57(3):1703–1715.
79. Akhtar M, Mahboob S, Sultana S, Sultana T. Pesticides in the River Ravi and its Tributaries Between its Stretches from Shahdara to Balloki Headworks, Punjab-Pakistan. *W E R.* 2014 Jan;86(1):13-9.
80. Maghsoudlou MA, Isfahani RB, Saber-Samandari S, Sadighi M. Effect of interphase, curvature and agglomeration of SWCNTs on mechanical properties of polymer-based nanocomposites: Experimental and numerical investigations. *Composites Part B: Engineering.* 2019 Oct 15;175:107119.
81. Alamri H, Low IM. Effect of water absorption on the mechanical properties of nano-filler reinforced epoxy nanocomposites. *Materials & Design.* 2012 Dec 1;42:214-22.
82. Razzaq, M. Y., Behl, M., & Lendlein, A. (2012). Magnetic memory effect of nanocomposites. *Advanced Functional Materials*, 22(1), 184-191.
83. Alomari A, Aldajah S, Hayek S, Moustafa K, Haik Y. Experimental investigation of the low speed impact characteristics of nanocomposites. *Materials & Design.* 2013 May 1;47:836-41.
84. Weigel TH, Mohr R, Lendlein A. Investigation of parameters to achieve temperatures required to initiate the shape-memoryeffect of magnetic

- nanocomposites by inductive heating. *Smart Materials and Structures*. 2009 Jan 13;18(2):025011.
85. Kumar, U. N., Kratz, K., Heuchel, M., Behl, M., & Leidlein, A. (2011). Shape-memory nanocomposites with magnetically adjustable apparent switching temperatures. *Advanced Materials*, 23(36), 4157-4162.
 86. Tarekegn MM, Hiruy AM, Dekebo AH. Nano zero valent iron (nZVI) particles for the removal of heavy metals (Cd^{2+} , Cu^{2+} and Pb^{2+}) from aqueous solutions. *RSC Adv*. 2021;11:18539–18551.
 87. Qasem NAA, Mohammed RH, Lawal DU. Removal of heavy metal ions from wastewater: A comprehensive and critical review. *npj Clean Water*. 2021;4:36.
 88. Behbahani TJ, Lan J, Abolghasemi H, Sadeghi MT, Fan J. Fe_3O_4 – FeMoS_4 : Promising magnetite LDH-based adsorbent for simultaneous removal of $\text{Pb}(\text{II})$, $\text{Cd}(\text{II})$ and $\text{Cu}(\text{II})$ heavy metal ions. *J Hazard Mater*. 2021;410:124560.
 89. Hoan NTV, Van HT, Khanh NM. Fe_3O_4 /reduced graphene oxide nanocomposite: Synthesis and its application for toxic metal ion removal. *J Chem*. 2016;2016:2418172.
 90. Liosis C, Papadopoulou A, Karvelas E, Karakasidis TE, Sarris IE. Heavy metal adsorption using magnetic nanoparticles for water purification: A critical review. *Materials*. 2021;14(24):7500.
 91. Mbuyazi TB, Ajibade PA. Magnetic iron oxides nanocomposites: Synthetic techniques and environmental applications for wastewater treatment. *Discover Nano*. 2024;19(1):158.
 92. Ge F, Li MM, Ye H, Zhao B. Facile fabrication of $\text{Fe}@\text{MgO}$ magnetic nanocomposites for efficient removal of heavy metal ion and dye from water. *Sep Purif Technol*. 2018;192:289–297.
 93. Li H, Chen J, Sun X, Zhang Y, Wang S. Magnetic biochar for heavy metal removal from wastewater: A review of recent advances and prospects. *Molecules*. 2023;28(15):5840.
 94. Mohammed MK. Synthesis and Characterization of $\alpha\text{-Fe}_2\text{O}_3$ Nanoparticles Using the Precipitation and Eco-Friendly Methods Resistant *Staphylococcus aureus* bacteria on the effect of extract *Tribulus terrestris* L. *Journal of Pharmaceutical Negative Results*. 2022 Oct 1;13(4).
 95. Rohaeti E, Yuliarto B. Reduction of silicon dioxide from bamboo leaves and its analysis using energy dispersive x-ray and fourier transform-infrared. *InIOP*

Conference Series: Earth and Environmental Science 2018 (Vol. 209, No. 1, p. 012048). IOP Publishing.

96. Hawezzy HJ, Qader AF, Omer RA, Ali LI. Magnetic nanoparticles for efficient heavy metal removal: synthesis, adsorption capacity, and key experimental parameters. *Reviews in Inorganic Chemistry*. 2025 ;45(3):587-96.
97. Almomani F, Bhosale R, Khraisheh M, Kumar A, Almomani T. Heavy metal ions removal from industrial wastewater using magnetic nanoparticles (MNP). *Appl Surf Sci*. 2020;506:144924.
98. Ahuja R, Gupta R, Sharma RK. Nano-modifications of biochar to enhance heavy metal removal from wastewater: A review. *ACS Omega*. 2022;7(49):44665–44681.
99. Joshi NC, Gururani P. Advances of graphene oxide based nanocomposite materials in the treatment of wastewater containing heavy metal ions and dyes. *Curr. Res. Green Sustain. Chem*. 2022;5:100306.
100. Khan SU, Khan MS, Qurashi A, Shah MA, Shahid M. Evaluation of Fe–Mg binary oxide for As(III) adsorption. *ACS Omega*. 2021;6:6217–6229.
101. Mohammed MK, Kareem SH, Abbas AS. Reduction of silicon dioxide from bamboo leaves and its analysis using energy dispersive X-ray and Fourier transform-infrared. *Plant Archives*. 2020;20(2):5309–5316.
102. Xu J, Li Z, Chen L, Wang J. Enhanced adsorption of heavy metals using amorphous MgO nanostructures. *J Chem Phys*. 2020;152(1):014702.
103. Arthy M, Phanikumar BR. Efficacy of Iron-Based Nanoparticles and Nanobiocomposites in Removal of Cr³⁺. *J. Hazard. Toxic Radioact*. 2016;20(3):04016005.