



Remediation of Aquatic Anionic Pollutants Using Magnetic Nanocomposites

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Abstract

Water contamination by anionic pollutants poses a significant threat to both public health and the environment. Effective treatment and recycling of this polluted water are essential to address the growing challenges of water scarcity and quality degradation. While conventional techniques like adsorption, ion exchange, chemical reduction, and membrane separation are employed for anion removal, they are often hindered by limitations such as high operational costs, the generation of secondary waste, and complex operational procedures. In recent years, iron oxide nanoparticles (IONPs) have emerged as a promising solution due to their remarkable adsorption capacity for anions. However, their high specific surface area leads to tendencies for agglomeration and oxidation, which subsequently reduces their reactivity and effectiveness. A key strategy to overcome these drawbacks is the incorporation of IONPs into supportive matrices such as graphene, chitosan, zeolite, polyaniline, or biochar. This process fabricates composite materials—like magnetic biochar, chitosan, or graphene—that enhance nanoparticle dispersion, prevent agglomeration, and improve stability, thereby maintaining high reactivity. Furthermore, these composites often inherit the beneficial properties of both the IONPs and the supporting matrix, leading to superior adsorption performance and easier magnetic separation from treated water. The primary aim of this review is to synthesize current knowledge and provide a comprehensive understanding of iron oxide-based composites, evaluating their potential as advanced materials for the efficient removal of anionic pollutants from water.

1 Introduction

Hazardous anionic contaminations is a group of priority pollutants which are present in surface water, groundwater soil environments in potentially harmful concentrations. Various potentially toxic anionic elements can cause potential threat to the aquatic and terrestrial environments and human health such as arsenic (As), chromium Cr(VI), fluoride (F), antimony (Desbonnet, #50) phosphate (PO_4^{3-}) and nitrate (NO_3), which are prevalent in wastewater and groundwater and must be treated before use of water for drinking, irrigation or discharge on the lands (John *et al.*, 2018).

In order to treat the contaminated water, various technologies have been evolved, such as lime softening, ion exchange, bioremediation, coagulation-flocculation, reverse osmosis (Hashim *et al.*, 2011), chemical reduction (An *et al.*, 2022), , membrane separation (Wang and Liu, 2021) and adsorption (Wang and Zhuang, 2019). From all these methods adsorption has several advantages compared to other methods such as improve efficiency, limited cost, ease of operation and reusability that reduces sludge disposal issues (Mahmood-ul-Hassan *et al.*, 2018; Xu *et al.*, 2018).

Nano-adsorbents is one of the adsorbent for the removal of anions from water. Among different types of nanoparticles magnetic iron oxide nanoparticles, are the most popular and

widely used in the removal of anions from water (Wei *et al.*, 2018; Shanableh *et al.*, 2020). However, unavoidable problems of nanoparticles such as air oxidation and agglomeration limit its application to remove anions from water (Wu *et al.*, 2017). It is thus necessary to prevent the degradation of ferromagnetic nanoparticles by forming protecting shells on the surface of nanoparticles (Simonsen *et al.*, 2018). Over recent year, strategies including magnetic graphene oxide (Su *et al.*, 2017), magnetic biochar (Yang *et al.*, 2017) and magnetic chitosan (Pradhan *et al.*, 2020) magnetic zeolite (Suazo-Hernández *et al.*, 2019) composite have been formed in order to transform iron oxide nanoparticles into advanced nanomaterials that are able to possess high adsorption capacity for anion, high reusability and fast recovery.

Considering global significance of anionic contamination in groundwater and surface water and their impacts on human health, it is imperative to critically analyze different relevant published data on removal of anions from water. In the following section, we will review the iron oxide (FeOx) nanoparticles (NPs) and highlight the recent developments of nano materials including graphene-based modified/ FeOx, magnetic biochar and chitosan modified FeOx-NPs, applied particularly in the water treatment of chromium, arsenic, fluoride, nitrate and phosphate removal from water sources.

2 Nanoparticles – a brief overview

Nanoparticles (NPs) are defined as fine particles having nanoscale external dimension or nanoscale internal structure ranging between 1 and 100 nm (ISO, 2015). They may be in the form of nanocomposites, nanofibers, nanotubes, nanoparticles and nanostructured materials. NPs can exist in liquid, solid and gaseous form. NPs also have 35-45% higher surface-area-to-volume ratios compared to atom or large particle (Jamkhande *et al.*, 2019)(ref????). This unique property resulted in higher specific surface area and it also influences different intrinsic properties such as strong surface reactivity which is size dependent (Auffan *et al.*, 2009). Also these NPs may vary according to their, chemical nature, size, shape, and, amorphous, crystalline, and solid-state of occurrence (Alvi *et al.*, 2021).

Various methods are used to synthesize NPs including physical, chemical and biological methods. Two chief approaches -top down and bottom up approaches have been used for the synthesis of nanoparticles. Previously top down approach was widely used which employed the transformation of bulk material into small nanosized particles. But such methodologies are ineffective to obtain a proper particle size and shape (Abid *et al.*, 2021). Contrarily, second one is the bottom-up approach for the synthesis of nanoparticles, which can make well-defined size, shape and chemical composition of NPs (Kolahalam *et al.*, 2019; Abid *et al.*, 2021). Typical examples are sputtering, chemical etching, explosion and laser ablation. Alternatively bottom-

up approaches result the formation of small particles from the crystalline areas that do not match with the perfect lattice. Typical examples are formation of nanoparticles by vapour deposition, sol-gel processes, electro-chemical deposition, bio-reduction and aerosol processes (Bhateria and Singh, 2019).

Moreover NPscan either be a zero dimensional where length, breadth and height fixed at single point, one dimensional which possess only one parameter, two dimensional which possess only two parameters (length and breadth) and three dimensional having all three parameters (length, breadth and height) (Ealia and Saravanakumar, 2017). These nanoparticles are further characterized by X-ray diffractometer (XRD), Brunauer–Emmett–Teller (Gamboal-Loira, #45), Fourier transform infrared spectroscopy (FTIR), Thermal gravimetric analysis (TGA), Photoluminescence spectroscopy (Yavuz, #99), Dynamic light scattering (DLS), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), NPs tracking analysis (NTA), Time of flight secondary ion mass spectrometry (ToF-SIMS), Atomic Force Microscopy (AFM), Electron diffraction, Electron backscatter diffraction (EBSD), Magnetic force microscopy (MFM) and X-ray magnetic circular dichroism (XMCD)

3 Iron oxides mineral-based nanoparticles

Iron oxides (FeOx) mineral nanoparticles have shown rapid development in nanoscience over the last ten years. Due to technological importance extensive studies have been carried out on the development of various synthetic route to yield FeOxNPs having desired characteristics (Roca *et al.*, 2019). Among FeOx-NPs mainly γ -Fe₂O₃ and Fe₃O₄ are extensively studied (Noqta *et al.*, 2019). In general FeOx are classified into different phases such as γ -Fe₂O₃ (ferromagnetic and maghemite), FeO (antiferromagnetic and wustite), α -Fe₂O₃ (weakly ferromagnetic or antiferromagnetic, hematite), Fe₃O₄ magnetite (Fe²⁺ Fe³⁺₂O₄, ferrimagnetic, superparamagnetic mostly when their size is less than 15nm) (Cornell and Schwertmann, 2003). Magnetite nanoparticles is one of the FeOx-NPs which is unique in its properties as it contains both divalent (Fe²⁺) and trivalent (Fe³⁺) arrangement of iron atoms. Therefore, magnetite has inverse spinal crystal structure in which half of the octahedral positions are occupied by Fe²⁺ and further lateral tetrahedral and octahedral positions are joined by Fe³⁺. Moreover magnetite nanoparticles exist as both p- and -n having a very minor band gap of 0.1 eV (Rioul *et al.*, 2016; Khan and Qurashi, 2018). Also magnetite nanoparticles have a great ability to adsorb heavy metals from a water system and can be easily recovered from the system with the assistance of external magnetic field ((El-Dib *et al.*, 2020; Zhang, J. *et al.*, 2020). Maghemite share a similar spinel structure to magnetite; however, maghemite has only Fe³⁺ present; for this reason maghemite is considered a fully oxidized magnetite. The

chemical formula of maghemite is often indicated as $\gamma\text{-Fe}_2\text{O}_3$ compared to Fe_2O_3 in order to differentiate it from another FeOx having same chemical formula and different crystalline structure (Devi *et al.*, 2019). It has a cubic and tetragonal system and its crystal habit is disks, cubes, rods, flowers and many others, such as hollow spheres, worms, stars or tetrapods in shape (Roca *et al.*, 2019).

Hematite is the second most common FeOx and is abundantly found in rocks and soils. It is iso-structural as corundum ($\alpha\text{-Al}_2\text{O}_3$) consisting of an intense structure connected with Fe^{3+} cation in octahedral coordination with oxygen in the hexagonal closed-packing system. The crystal system of hematite is hexagonal with the Miller parameters of $c = 13.752 \text{ \AA}$ and $a = 5.0346 \text{ \AA}$. The structure can be depicted as stacking of Fe^{3+} ion sheets between two layers of oxygen in the closed-packed system, holding together by covalent bond. The structure was shown in a three dimensional framework along with trigonally distorted octahedral FeO_6 associated with thirteen neighbors by one face, six vertices and three edges. Because iron is in trivalent state (ferric, Fe) each one of the oxygen is linked with just two Fe ions and so, just two of three available oxygen octahedron are occupied. This distinctive framework tends to make the structure neutral without any deficit or excess in charge (Cornell and Schwertmann, 2003). Goethite is a another widespread FeOx mineral having an orthorhombic unit cell consisting of hexagonally close-packed array of O^{2-} and OH^- anions with Fe^{3+} cations occupying half of the octahedral sites (Liu *et al.*, 2014). Moreover, unit cell parameters of goethite are $a = 9.95 \text{ \AA}$, $b = 3.01 \text{ \AA}$, $c = 4.62 \text{ \AA}$ (Testa-Anta *et al.*, 2019). In goethite each iron atom is octahedrally coordinated by three hydroxyl group and three oxygen atoms. Two Fe atoms in the center of the unit cell are edge sharing, and each of those Fe atoms shares a corner with an additional Fe atom at the top right and bottom left corners of the unit cell. When extended in three dimensions, all Fe atoms share edges with Fe atoms in neighboring unit cells, forming chains parallel to the c-axis (Smith *et al.*, 2015). Goethite can be found in different shapes and morphology; however its morphology is essentially acicular or needle-like (Shaw *et al.*, 2011). Wustite is another phase of FeOx has a cubic rock-like structure with the octahedral arrangement of iron and oxygen in unit cell (Boudjemaa *et al.*, 2015). Also the wustite (Feo) is considered of special interest from geophysical view point because it is a constituent of the lower mantle of earth. Moreover this FeOx has melting point near 1400°C when an oxygen pressure is in the range of 10^{-25} to 10^{-6} bar and its steady state temperature is near 600°C (Gavarri and Carel, 2019). Iron and oxygen planes of wustite form two-dimensional hexagonal lattice having an inter-atomic distance of $3.04 \text{ \AA}$ (Ramimoghadam *et al.*, 2014). Akaganeite ($\beta\text{-FeOOH}$) is the other iron oxohydroxide phase which exhibit large tunnel structure consisting of strongly bonded iron atoms and impurities of Chloride ions. Iron hydride (FeH) and Chloride ions present in octahedral site

and H_2O is assumed to be present in the tunnel of framework (Jassal *et al.*, 2016).

4. Properties of anionic pollutants in water

Arsenic (As) is an extremely hazardous metalloid gaining global concern (Ahmed, Minhaz Farid *et al.*, 2021; Wu *et al.*, 2021). Contamination of groundwater with As has occurred in many regions of the world including India, China, Pakistan, Mexico, Bangladesh and the United States as a result of various natural and anthropogenic processes (Sarkar and Paul, 2016; Siddiqui *et al.*, 2019). The groundwater in these regions can contain As concentrations more than $50 \mu\text{g/L}$ to ????? (Smedley, 2005; Siddiqui *et al.*, 2019). Over 200 million people around the world are at risk of arsenic poisoning due to ingestion of arsenic-contaminated drinking water (Shakoor *et al.*, 2015; Shahid *et al.*, 2018; Shakoor *et al.*, 2018)?, which is an important pathway of an As exposure to humans. Many As compounds can cause health problems such as lung, skin, bladder and kidney cancers (Gamboa-Loira *et al.*, 2017). So due to toxic As nature drinking water maximum permissible limit $10 \mu\text{g L}^{-1}$ have been set by World Health organization (WHO) and the United States Environmental Protection Agency (US-EPA) (Environmental Protection Agency, 2016; World Health Organization, 2017) As is ubiquitous element with several different chemical forms have varying degrees of toxicity. In addition the distribution of arsenic in water system can increase with pH and redox potential (Zakhar *et al.*, 2018). As normally present in four oxidation states As^{3-} (arsine) As^0 (elemental arsenic), As^{3+} (arsenite) and As^{5+} (arsenate) (Beiyuan *et al.*, 2017; Cui *et al.*, 2018; Marc *et al.*, 2018). Higher level of As^{3+} were found in ground water under anaerobic condition while in aerobic oxygen rich habitat As, is present in the form of arsenate (Uppal *et al.*, 2019). In addition, there are organic As type whose toxicity are less than inorganic arsenic types these organic arsenic include dimethylarsinic acid and monomethylarsonic (González-Martínez *et al.*, 2018). Fluorine, the first member of the halogen family of element is another most electronegative and highly reactive element (Ighalo *et al.*, 2021) and occur in its ionic form as fluoride in water (Ali *et al.*, 2019). It is the richest trace element and is the 13th most abundant element in the earth crust at about 0.3g/kg (Habuda-Stanić *et al.*, 2014). Fluoride can be beneficial to living organism only when their concentration is $<1.5\text{mg/L}$ and further its acceptable limit is 1mg/L (Ahmad *et al.*, 2019). Also it is found in all natural water but its concentration increases due to anthropogenic activities such as industrial waste water (steel, slag, iron, and aluminum) or agricultural activities such as phosphate fertilizer and certain pesticides (Gupta *et al.*, 2015; Srivastav *et al.*, 2018; Maurya *et al.*, 2019). Various places in worldwide exceed the desired level of fluoride including China, UK, Argentina, Spain, Middle East, Pakistan, Italy, West Indies, Mongolia, Norway, Netherlands, Poland (Ali *et al.*, 2016). Moreover, long-term consumption of water with excessive fluoride significantly increase the risk of dental and

skeletal fluorosis. However, this anion also affected the soft tissue and cause non-skeletal fluorosis (Patil *et al.*, 2018). In addition it can even cause dyspnea, salivation, muscle weakness, gastroenteritis, restlessness, anorexia, sweating, ventricular abnormalities and tachycardia (Sahu *et al.*, 2017).

Phosphorus is another anion widely used in modern agriculture and chemical industry, which generally present in aqueous solution in the form of phosphate. This phosphate (PO_4^{3-} , HPO_4^{2-} , H_3PO_4 , H_2PO_4^-) pollution is the main cause of algal blooms and eutrophication, which have disastrous effects such as worsening of the aesthetic appearance of water, depletion of dissolved oxygen and production of toxins by harmful algae (Al Jaber *et al.*, 2019). To avoid the acceleration of phosphate pollution, United States Environmental Protection Agency has established a recommended limit of 0.01mg/L of total phosphate for reservoirs and lakes (Kumar, P.S. *et al.*, 2019). Moreover, phosphate is mainly derived from non-renewable energy source and it is estimated that reserves of these phosphate will last within the next 100 to 400 years due to an annual demand growth rate of 3% (Zhang, Z. *et al.*, 2020).

Hexavalent chromium is also an emerging contaminant due to its toxicity and become one of the world's most serious environmental concern. This hexavalent Cr [Cr(VI)] generated from variety of industrial operations like paint, textile, leather, metal finishing, pigments and electroplating industries which severely damage the aquatic ecosystem, if left untreated (Zare *et al.*, 2018). Moreover, Cr(VI) exists in aquatic system in many different toxic forms such as dichromate, divalent chromate, or monovalent chromate which act as powerful oxidizing agents Kumar, H. *et al.* (2019). This in turn affect human health through food chain poisoning and ground water pollution. Exposure to Cr(VI) through dermal, respiratory and oral pathway cause cancer and severe illness (Liu and Yu, 2020). Also, the metal is perilous mutagenic agent that cause cell transformation and genotoxicity (do Nascimento Monteiro *et al.*, 2018). In terms of Cr(VI) concentration regulation, the World Health Organization (WHO) and U.S. EPA recommend a MCL of 50 µg/L for Cr(VI) in drinking water (Bhaumik *et al.*, 2012; Lin *et al.*, 2019).

Nitrate is ubiquitous pollutant and is found everywhere in ground water and surface water because of its high solubility. The excessive nitrate in potable water than the allowable concentration (15mg/L) according to World Health Organization (WHO) induces negative consequences on both in human health (reproductive damage, blue baby syndrome, gastric cancer, abortions, mouth ulceration, damage to stomach lining and increased risk of methemoglobinemia) and natural ecological environment including creation of eutrophication (Hashim *et al.*, 2017; Paladino *et al.*, 2018; Wu, J. *et al.*, 2018; Wu *et al.*, 2019).

5. Available technologies for the removal of anions from water

Currently there are several methods available for anion removal such as ion exchange, biological denitrification (Pang and Wang, 2020), flocculation, adsorption, coagulation and membrane filtration (Nicomel *et al.*, 2016; Mohanty, 2017). Despite the success of these methods for removal of anions, they do face certain disadvantages like cost or management of sludge. Each of these methods is associated with shortcomings such as chemical treatment (coagulation/precipitation) need equipment for remediation of waste water and also large amount of sludge produced at the end of this process (Poursaberi and Hassanisadi, 2013). On the other hand reverse osmosis need the application of membrane which add extra burden to operation and capital cost (Payne and Abdel-Fattah, 2005). Among the possible techniques for treatment of water, the adsorption process provide flexibility in design and operation and also produce low amount of sludge or toxic intermediates (Pourebrahim *et al.*, 2017). Now a days different adsorbent materials have been developed to adsorb anions (Wong *et al.*, 2016; MENG *et al.*, 2017) such as activated carbon (Kyriakopoulos and Doulia, 2006; Mondal *et al.*, 2007), hydrotalcite-like compounds and activated alumina (Tripathy and Raichur, 2008; Cai *et al.*, 2012) but their poor adsorption ability and difficult separation limit their practical applications (Kyriakopoulos *et al.*, 2003). Hence, the development of more efficient process for the removal of anions is important. Iron based adsorbent have been extensively studied and it showed prominent anion removal efficiency (Cutler *et al.*, 2014; Chen *et al.*, 2015). Various type of nano-adsorbent have been developed recently i.e. functionalized FeOx-NPs for the efficient removal of various toxic anions from wastewater because of its inexpensive preparation, large surface area and low toxicity (Neto *et al.*, 2019; Subedi *et al.*, 2019; Altun and Ecevit, 2020). However, these nanoparticles are easily agglomerate and oxidized because of their high specific surface area which further reduce their reactivity (Ravikumar *et al.*, 2016; Xie *et al.*, 2017; Song *et al.*, 2022). To overcome these limitations researchers have used modified FeOx-NPs to exhibit better reactivity (Tang *et al.*, 2021). So, chitosan, biochar and graphene have gained much interest to modify these FeOx-NPs because of their beneficial features such as of higher surface area, non-toxicity, wide range of sources and low price (Wei *et al.*, 2018; Huang *et al.*, 2020; Keshvardoostchokami *et al.*, 2021). These modified magnetic nanoparticles were then commonly used for efficient removal of anions due to their strong adsorption and also easy separation of these magnetized nanoparticles by using external magnetic field (Yavuz *et al.*, 2006; Badrudoza *et al.*, 2013; Gallios *et al.*, 2017).

6. Iron oxide mineral nanoparticles for removal of anionic contaminants of priority concern

6.1 Magnetic nanoparticles modified chitosan composite

Chitosan, a chitin derivative by N-deacetylation, is the second most abundant natural polymer in the world (Tanhaei *et al.*, 2020). This natural polymer has large number of

hydroxyl and amino groups on the surface, which form stable chelates with heavy metals and dyes (Abri *et al.*, 2019; Alaswad *et al.*, 2020; Sadiq *et al.*, 2020). However, chitosan has low mechanical strength and poor solubility in neutral aqueous solution which further limits its application in aqueous solution (Vakili *et al.*, 2014; Yang *et al.*, 2020). Moreover, chitosan in its natural form of flaked has no porosity and low specific area which limits its adsorption capacity (Miretzky and Cirelli, 2011). Also, amino and hydroxyl functional group of natural chitosan are not readily available for anionic toxic elements such as for F it was reported to be 0.052 mg g⁻¹ (Viswanathan and Meenakshi, 2010).

Chemical modification by magnetic Fe oxide nanoparticles can effectively avoid the chemical weakness of chitosan for anionic contaminants removal from water. Chitosan can be mixed with FeOx-NPs to remove anionic pollutants. As chitosan is an essential ingredient to remediate the polluted water and their low cost, nontoxic nature, effective functional group and their macromolecular structure make chitosan become a unique adsorbent (Mohamed *et al.*, 2020). Moreover, the magnetic nanoparticles would provide the magnetic characteristics to composite which help to remove magnetic nanoparticle after treatment by applying a magnetic field, after this solution can be easily filtered by simple filtration technique (Neeraj *et al.*, 2016)..

The majority of research on anionic PTEs has been performed on arsenic by using FeOx magnetic NPs from water. For instance, Ayub *et al.* (2020) evaluated arsenite (As(III)) and arsenate (As(V)) removal by magnetic chitosan biosorbent beads (MCBB) from arsenic-contaminated water. Sorption capacity of a As (Reigstad, #51) and As (V) by magnetic chitosan biosorbent was 73.69 mg g⁻¹ and 79.49 mgg⁻¹ under specific conditions. The authors also observed that sorption experiment was pH dependent and maximum adsorption of As (Reigstad, #51) and As (V) was observed at pH 6.7. Removal percentage of As (V) and As (Reigstad, #51) by MCBB (1.5 and 2 g/L) under the pH of 6.7 was 99.5 and 99% respectively. Ahmed, Mian Fawaz *et al.* (2021) also studied the removal of arsenic from contaminated water by using chitosan and hybrid beads of FeOx-NPs. They synthesized the hybrid bead with three different concentration of nanoparticles that is FeCh-60, FeCh-40 and FeCh-20. They observed that maximum adsorption capacity (81%) of As is achieved in 10hr under the condition of pH 7.4. The adsorption isotherms followed Langmuir model and the best fitted model was pseudo-second-order.

Liu *et al.* (2015) synthesized magnetic chitosan nanoparticles and evaluated their adsorption capacity for As(V) and As (Reigstad, #51). The results showed that highest adsorption capacities for As (V) and As(Reigstad, #51) are 66 mgg⁻¹ and 60 mgg⁻¹ at pH 6.8, respectively. Wang *et al.* (2014) prepared magnetic impregnated chitosan bead for the removal of As (V) and As (Reigstad, #51) from

water. The adsorption isotherms followed Langmuir model and the best fitted model was pseudo-second-order and the maximum adsorption capacity for As(V) and As(Reigstad, #51) are 35.7 mgg⁻¹ and 35.3 mgg⁻¹ at pH 6.8, respectively. Furthermore, effects of different factors such as arsenic ion concentration, pH, adsorbent dosage and presence of other anion were also observed systematically. On the other hand, Gogoi *et al.* (2017) evaluated As(V) sorption by chitosan coated magnetite nanoparticle from contaminated water. In this research the adsorption isotherms followed Langmuir model and the best fitted model was pseudo-second-order and the maximum adsorption capacity for As(V) is 10.81 mgg⁻¹ at room temperature and normal pH. Furthermore, considering the contact time of 120min and an adsorbent dosage of 1g/L, the adsorption capacity related to initial As(V) concentration (0.6mg/L) was reduced to less than 0.01mg/L.

Kloster *et al.* (2020) also studied the As(V) adsorption on chitosan with magnetic nanoparticles. These authors observed that chitosan with magnetic nanoparticles presented higher adsorption capacity for As(V) compared to capacity of neat chitosan. They also observed that chitosan made with FeOx-NPs showed good As(V) (10.4 mgg⁻¹) adsorption capacity compared to neat chitosan film. As removed by neat chitosan film was 1.6 mgg⁻¹ which was six time less than the As removed by chitosan with magnetic nanoparticles.

These magnetic chitosan based composite also considered to be excellent candidate for fluoride removal because of its easy separation and high chelating capability (Hu *et al.*, 2018). Dehghani *et al.* (2020) reported the synthesis and use of magnetic chitosan composite for fluoride removal. The optimum sequestration efficiency of fluoride (93.1%) was noticed at an adsorbent rate of 1mg/L, contact time of 25min and pH of 3. From the experimental data reported in this study, the sorption process of fluoride followed the Langmuir and pseudo 2nd order kinetic model. (Mohseni-Bandpi *et al.*, 2015) Mohseni-Bandpi *et al.* (2015), prepared novel magnetite-chitosan composite for the confiscation of fluoride ions from drinking water. Finding indicated uptake capacity of fluoride ion was 9.43 mgg⁻¹ at optimum temperature of 50°C and pH=7. Thermodynamics results also show that the sorption procedure of fluoride ion was feasible, spontaneous and endothermic.

The sorption of Cr(VI) onto nano iron oxides impregnated chitosan composite from water was examined by (Lu *et al.*, 2021). Sorption capacity of Cr(VI) onto synthesized composite reached 92.04 mgg⁻¹ at pH 5, 20°C. Furthermore, this composite showed bi-function mechanisms (sorption / reduction) for Cr(VI) removal from aqueous solution. They also observed that the existence of coexisting ions (PO₄³⁻ and SO₄²⁻) have a significant effect on the adsorption of Cr(VI). Samrot *et al.* (2019) used stable chitosan-coated Super-paramagnetic FeOx-NPs for removal of Cr(VI) from water. The results exhibited that the

maximum adsorption capacity reached 50 mgg^{-1} by using 10 g/L adsorbent at a pH of 2. In this research adsorption isotherm followed Langmuir, Temkin and Freundlich model. Bavasso *et al.* (2019) utilized the chitosan-magnetite nanocomposite for the removal of Cr(VI) from aqueous solution. The results exhibited that the optimal pH value was 3, the removal percentage reached 62% for 20 mg/L of Cr(VI) initial concentration within 120 min at 45°C . Similarly chitosan-coated magnetite nanoparticles was used as an adsorbent for the removal of Cr(VI) ions. The obtained results indicated that chitosan-coated magnetite nanoparticles was a highly efficient adsorbent for the removal of Cr(VI) ions from laboratory wastewater at pH 2. In this experiment the result of the adsorption were described in the Langmuir isotherm model where the concentration range of Cr(VI) was 15.36 mgg^{-1} (Sanchayanukun and Muncharoen, 2019). Ravi and Anuradha Jabasingh (2018) also evaluated the effectiveness of chitosan-coated magnetic adsorbent for Cr(VI) from aqueous mixture. Removal of Cr(VI) was studied through batch adsorption experiment was carried out at pH 5 under equilibrium condition. Under optimum conditions, the results showed that the removal of Cr(VI) from aqueous mixture was 96.3%. Moreover, the equilibrium adsorption data fitted very well with Langmuir isotherm model and pseudo first order kinetic model. Subedi *et al.* (2019) prepared chitosan modified ferrites by solvothermal process for Cr(VI) removal. The adsorption capacity of Cr(VI), as determined from the Langmuir adsorption isotherm in batch experiment, was 142.32 mgg^{-1} . Moreover, the adsorption kinetics fitted well with pseudo-second-order model. Additionally, an important fact is that this composite could be used several times making the process economically feasible.

Moreover, various researchers have recommended the use of this composite for phosphate removal from wastewater. For instance, Kim *et al.* (2018) fabricated iron oxide nanoparticle-loaded chitosan composite for phosphate removal from natural water. It was found that maximum percent removal capacity of magnetic chitosan towards total phosphorus was 52.3% with a phosphate removal capacity of 0.059 mg P/g . Also, it was suggested that these composites was not sensitive to pH change at pH value of 5-9, demonstrating that adsorption of phosphate would not be varied at neutral pH. In attempt Shanableh *et al.* (2020) also scrutinized the removal of phosphorus by chitosan-coated nanoscale zero-valent iron. They examined that pristine nanoparticle exhibited good adsorption capacity (437.4 mgg^{-1}) for phosphate compared to chitosan-coated nanoscale zero-valent iron (289.2 mgg^{-1}) at pH 5. Contrariwise, at higher pH (7) the adsorption of phosphate decreased in both composite and pristine nanoparticles. The experimental result pointed out that phosphate removal by zero-valent nanoparticles depend on both precipitation and adsorption processes (Nagoya *et al.*, 2019). Precipitation occurs due to release of Fe^{2+} , and corrosion of Fe^0 resulting in the formation of Fe_3

$(\text{PO}_4)_2$ and FePO_4 in aqueous solution. In case of adsorption, it depend on isoelectric point of zero-valent iron isoelectric point (isoelectric point of these nanoparticles are in between 6 to 9). So, when the $\text{pH} \leq$ isoelectric point sorption of phosphate is favored on positively charged zero-valent iron surface. In another word at $\text{pH} > 5$ adsorption process contributed more significantly compared to process of precipitation. Indeed phosphate is polyprotic and exist in different species at different pH in solution such as H_3PO_4 ($\text{pH} < 2$), H_2PO_4^- ($\text{pH} 2-7$), HPO_4^{2-} ($7-12.5$) and PO_4^{3-} (> 12.5) (Karthikeyan and Meenakshi, 2020). At higher pH the adsorption of phosphate ions decreases due to competition and diffusion resistance effect of hydroxyl ions that occupied the adsorption sites (Zhang, B. *et al.*, 2018). Moreover, Fe^{3+} played an important role in adsorption process because it might affect the change of pH in solution. A large amount of hydrogen ions are produce during the hydrolysis reaction of Fe^{3+} which further enhance the protonation process under acidic condition (Liu *et al.*, 2016). In another investigation, carboxylated chitosan modified ferromagnetic nanoparticles have been used for the removal of phosphate anions from aqueous solution. They also observed that maximum adsorption of phosphate was achieved at $\text{pH}=3$ (Mohammadi *et al.*, 2019).

An *et al.* (2022) in their study, combined the chitosan gell balls and micro-sized zero-valent iron, aiming at synthesizing a new adsorbent for nitrate removal. At influent nitrate concentration 15 mg/L , $\text{pH}= 7$, 25°C , the nitrate removal rate was found to be 0.37 mg/L , which was 11.33 times higher than that of pristine zero-valent iron. Moreover, they observed that the formation of dense iron-oxide crystal on the surface of composite reduces due to the formation of amorphous chelates. This further helps the reaction to occur effectively and continuously and eliminates the passivation phenomena. The elimination of this passivation phenomena reduces the sensitivity of this magnetic chitosan composite to pH. Another novel strategy has been employed for the adsorptive removal of nitrate by carboxylated chitosan modified with ferromagnetic nanoparticles. Moreover, the adsorption process followed the Langmuir isotherm model with R^2 value of 0.9999 which was much better than R^2 value obtained from Freundlich isotherm model. Kinetic model was well fitted to Ho and McKay's model which suggested that the rate controlling step was chemisorption in nature (Mohammadi *et al.*, 2019).

6.2 Graphene-based nanoadsorbent:

Graphene is a monolayer of carbon atom film tightly packed in a two-dimensional structure forming a honeycomb lattice (Vinayan, 2016). This sort of 2D atomic crystal structure renders graphene an extraordinary material with multifunctional properties such as larger specific surface area ($\sim 2630 \text{ m}^2 \text{ g}^{-1}$), high thermal conductivity, strong mechanical strength, electronic and optical properties, which attracted attention of scholars from different background

(Service, 2009). Graphing seems to be an ideal sorbent due to its good chemical stability and ultrahigh surface area (Soltani *et al.*, 2018; Yang *et al.*, 2018). In spite of this, graphene is hydrophobic in nature and difficult to dissolve in polar solvent so it is difficult to make composite with other materials and further it limits its practical application. As one of the important derivatives of graphene is graphene oxide (GO). These graphene oxide (GO) and reduced graphene oxide (rGO) are evaluated as promising new materials for arsenic removal from waters and wastewater due to their high thermal conductivity, mechanical stability, high surface area, and surface functional groups that provide a very good support for different compounds in arsenic adsorption (Wang *et al.*, 2013; Wu, L.-K. *et al.*, 2018). However, good dispersive property of graphene oxide becomes a major obstacle to separate it from aqueous phase even after adsorption of heavy metal. To address this flaw, magnetization of GO is the best solution whereby using the external magnetic field, magnetized GO can be easily separated (Lingamdinne *et al.*, 2019). For instance crystalline magnetic iron oxide nanoparticles have been loaded on reduced graphene oxide (RGO) and graphene oxide for the adsorption of arsenic (Chandra *et al.*, 2010; Ding *et al.*, 2015). Moreover, Pradhan *et al.* (2020) observed the removal efficiency of arsenic (V) up to 96% for contact time 120 min at pH 7 by iron oxide embedded reduced graphene oxide. Even Magnetic iron oxide/graphene oxide (MGO) could give a maximum adsorption capacity of 54.18 and 26.76 mgg^{-1} for As(Reigstad, #51) and As(V) species in an aqueous medium (Yu *et al.*, 2015).

Chandra *et al.* (2010) developed the 10nm Magnetite-Reduced Graphene oxide (rMGO) composite for adsorption of arsenic. They observed that (rMGO) composite had adsorption capacity over 99% for both As(Reigstad, #51) and As(V) species. Sherlala *et al.* (2019) employed a magnetic graphene oxide composite for magnificent adsorption of arsenite from aqueous solution. Here, high arsenite removal efficiency (> 99.95%) was observed at adsorbent dosage of 0.3g/L, contact time 77min and at pH 7. In another study (Su *et al.*, 2017) conducted an experiment to investigate the adsorption behavior of iron oxide-Graphene oxide nanocomposite for removal. They demonstrated that maximum adsorption capacity of iron oxide-Graphene oxide nanocomposite for As(Reigstad, #51) and As(V) were 147 and 113 mgg^{-1} , respectively. There are numerous applications of magnetic iron oxide composites with graphene for the removal of As from water that are summarized in **Table 2**.

The sorption of fluoride onto FeOOH-graphene oxide nanocomposites from water was examined by Kuang *et al.* (2017), using batch adsorption experiment. FeOOH-graphene oxide nanocomposites revealed an efficient adsorption behavior towards fluoride ions at which $q_{\text{max}} = 17.67 \text{ mgg}^{-1}$ at pH range of 2.11-10.39 in 8hrs. Furthermore, studying the impact of coexisting anions (SO_4^{2-} , PO_4^{3-} , NO_3^- , Cl^- , SiO_3^{2-} and HCO_3^-) on the adsorption efficacy pointed out

that fluoride ions fiercely compete with HCO_3^- ions for binding site of nanocomposite. This HCO_3^- ions decreased the adsorption of fluoride ions by 29.6%. In an experiment by Fan *et al.* (2016), goethite anchoring regenerated reduced graphene oxide synthesized by facile one-step hydrolysis pathway have been used for the removal of fluoride from drinking water. The maximum adsorption capacity of this composite was 24.67 mgg^{-1} at 25°C. Furthermore, X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR) proved that the uptake of fluoride by this composite was governed by ion-exchange method. The fluoride will exchange with sulfate group of goethite crystal structure and releases hydrogen ion to solution. Liu *et al.* (2016), experimented for the adsorption of fluoride by using akaganeite anchored graphene oxide nanocomposite. The maximum adsorption capacity of this composite was 17.67 mgg^{-1} at room temperature. Moreover, the equilibrium adsorption data fitted very well with Langmuir isotherm model and pseudo second order kinetic model.

(Sarikhani and Manoochehri (2020) synthesized novel magnetic graphene oxide nanocomposite for Cr(VI) removal from aqueous environment. The prepared nanocomposite are characterized by X-ray diffraction (XRD), FTIR, Vibrating sample magnetometry (VSM) and Scanning electron microscopy (SEM). The equilibrium data was fitted to Langmuir isotherm model and the maximum adsorption capacity of Cr(VI) on the nanocomposite was 250 mgg^{-1} . Additionally, the authors stated that the adsorption process followed a pseudo-second-order model. Similarly the research conducted by Neolaka *et al.* (2020) reported that graphene oxide-magnetic ($\text{GO-Fe}_3\text{O}_4$) composite with a high specific surface area have effectively removed Cr(VI) from water. The adsorption data obeyed the Langmuir equation with the maximum adsorption capacity of 3.197 mgg^{-1} at pH 2 with contact time for 80min and temperature of 298K for Cr(VI). The adsorption kinetics followed pseudo-second-order kinetics having high regression coefficient (> 0.99). The research by Khare *et al.* (2018) also tested the removal of Cr(VI) by graphene coated maghemite nanoparticles. The removal of Cr(VI) was pH dependent and maximum removal of this ion was obtained at pH 2 when the equilibrium time was 15min. Also in this research Cr(VI) adsorption agreed well with pseudo-second-order kinetics and Freundlich model isotherm with the maximum adsorption capacity of 352.1126 mgg^{-1} .

The research conducted by Rajput *et al.* (2016) reported that magnetic magnetite nanoparticles with a high specific surface area effectively removed Cr(VI) from water. In this research equilibrium data were described by Langmuir, Freundlich, Radke and Sips, Toth, and Redlich-Peterson isotherm models. The Sips and Langmuir adsorption isotherm best described the Cr(VI) adsorption on magnetic magnetite nanoparticles with the adsorption capacity of 34.87 mgg^{-1} . Harijan and Chandra (2016) also proved that magnetic

Table 2 Adsorption capacities and experimental conditions of novel magnetic graphene/graphene oxide and nano-magnetic chitosan (nM-Cs) for the removal of anions from water

Adsorbent	Anion species removal	Characterization	Reagent	Isotherm - model	Kinetic model	pH	Adsorption capacity (mg/g)	References
nM-Cs	As III As(V)	SEM, FTIR, XRD	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	6.7	73.69 79.49	(Ayub <i>et al.</i> , 2020)
nM-Cs	As(V)	SEM, FTIR	Fe ³⁺ /Fe ²⁺	Freundlich	Pseudo - second-order model	-	10.4	(Kloster <i>et al.</i> , 2020)
nM-Cs	As(V)	FTIR, SEM, XRD, TEM	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	6.7	10.81	(Gogoi <i>et al.</i> , 2017)
nM-Cs	As III As(V)	—	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	6.8	60.2 65.5	(Liu <i>et al.</i> , 2015)
nM-Cs	F ⁻	XRD, FTIR, TEM	Fe ₃ O ₄	Freundlich	Pseudo - second-order model	7	9.43	(Mohseni-Bandpi <i>et al.</i> , 2015)
nM-Cs	F ⁻	XRD, FTIR, DLS	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	3	0.288	(Mohammadi <i>et al.</i> , 2019)
nM-Cs	Cr(VI)	UV-Vis, SEM, XRD, FTIR, EDX, VSM, AFM	Fe ₃ O ₄	Freundlich	-	2	50	(Samrot <i>et al.</i> , 2019)
nM-Cs	Cr(VI)	FTIR, SEM, XRD, BET	Fe ₃ O ₄	Freundlich	Pseudo - second-order model	2	142.32	(Subedi <i>et al.</i> , 2019)
nM-Cs	Cr(VI)	XRD, FTIR, XPS, FETEM, TGA-DSC	β-FeOOH α-Fe ₂ O ₃	Freundlich	Pseudo - second-order model	5	92.04	(Lu <i>et al.</i> , 2021)
nM-Cs	P ⁻¹	SEM, XRD	Fe ²	Langmuir	Pseudo - second-order model	5	289.2	(Shanableh <i>et al.</i> , 2020)

nM-Cs	P ⁻¹	XRD, DLS	FTIR,	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	5	4.604	(Mohammad i <i>et al.</i> , 2019)
nM-Cs	NO ₃ ⁻	XRD, DLS	FTIR,	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	3	0.247	(Mohammad i <i>et al.</i> , 2019)
MGO	As III As(V)	XPS, XRD	SEM,	Fe ₃ O ₄	The Sips isotherm	Pseudo - second-order model	7	38 14	(Yoon <i>et al.</i> , 2017)
MGO	As III As(V)	SEM, XPS, XRD	EDS,	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	7	85 37	(Yoon <i>et al.</i> , 2016)
rMGO	As III As(V)							57 12	
rMGO	F ⁻	XRD, SEM, TEM		α -FeOOH	Langmuir	Pseudo - second-order model	7. 2	24.67	(Fan <i>et al.</i> , 2016)
MGO	F ⁻	XRD, TEM	SEM,	β -FeOOH	Langmuir	Pseudo - second-order model	-	17.67	(Iiu <i>et al.</i> , 2016)
MGO	Cr(VI)	XRD, SEM, EDX	FTIR,	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	2	3.197	(Neolaka <i>et al.</i> , 2020)
MGO	Cr(VI)	FTIR, XRD, TEM, RM	LIBS, SEM,	Fe ₂ O ₃	Freundlich	Pseudo - second-order model	2	352.1126	(Khare <i>et al.</i> , 2018)
MG	Cr(VI)	XRD, TEM	FTIR,	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	6. 6	16.8	(Harijan and Chandra, 2016)
MGO	P ⁻¹	XRD, TEM	FTIR,	β -FeOOH	Langmuir	Pseudo - second-order model	7	45.2	(Harijan and Chandra, 2017)
rMGO	NO ₃ ⁻	XRD, SEM, TEM	FTIR,	Fe ₃ O ₄	Langmuir	Pseudo - second-order model	7	48.15	(Pu <i>et al.</i> , 2019)

graphene composite is an efficient sorbent for Cr(VI) removal from water solution. The adsorption data on magnetite graphene composite well fitted with Langmuir model indicated that adsorption process is governed by monolayer adsorption with maximum sorption capacity 16.8 mgg^{-1} .

Recent developments include the successful removal of phosphate by graphene oxide decorated with Fe_2O_3 nanoparticles using ferric chloride as a source of iron (Bai *et al.*, 2018). It was observed that this composite exhibited maximum adsorption capacity of 93.28 mgg^{-1} , in 50 mg/L phosphate solution at pH 6. The obtained graphene oxide composite has also has fast kinetics in which the equilibrium was reached within 5min. Also, the adsorption capacity of phosphate was very stable in the studied pH range (2-10.5) and temperature range of 293-333K. In another study, Akram *et al.* (2019) magnetic graphene oxide composite for high performance adsorption of phosphate in aqueous solution. The kinetics and isotherm model were followed by pseudo-second order and Freundlich model with maximum adsorption capacity of 135.3 mgg^{-1} . Moreover, Harijan and Chandra (2017) synthesized akaganeite nanorods decorated graphene oxide sheet for phosphate removal from aqueous solution. The maximum phosphate removal capacity was found for 5wt% loading of graphene oxide onto akaganeite. This loading further increases BET surface area of akaganeite from 20 to $80 \text{ m}^2/\text{g}$ and its aspect ratio with a corresponding maximum adsorption capacity of 45.2 mgg^{-1} at pH 7 and room temperature 30°C . In another attempt, Tran *et al.* (2015) examined the removal of phosphate from water by engineered graphene nanoparticles composite. This composite showed a superb adsorption capacity and remove phosphate up to 350 mg/L . Besides, the adsorption isotherm data was better described by Freundlich isotherm model and pseudo-second order kinetic model.

In recent year nano zero-valent nanoparticles has been extensively used for removal of nitrate from wastewater due to their active properties (Ribas *et al.*, 2017; Zhu, F. *et al.*, 2018). However, the shortcomings of nano zero-valent iron (instability and particle agglomeration) limits its practical application (Lv *et al.*, 2014). Previous researchers, have performed many studies for the modification of nano zero-valent iron with graphite-oxide. For instance, Pu *et al.* (2019) investigated the removal of nitrate by reduced graphite oxide supported nano zero-valent iron. They found that these composite reached $Q_{\text{max}} = 48.15 \text{ mgg}^{-1}$ at optimum conditions including 1 g of the adsorbent dosage and optimum pH 7. Furthermore, studying the impact of coexisting anions (SO_4^{2-} , Cl^- and HCO_3^-) on the adsorption efficacy pointed out that nitrate ions fiercely compete with HCO_3^- ions for binding site of nanocomposite. This HCO_3^- ions decreased the adsorption of nitrate ions by 100%. Similar type of nano zero-valent iron-reduced graphene oxide have been used for the removal of nitrate from aqueous solution (Khoshro *et al.*, 2020). This composite reached 96% nitrate removal capacity

only after 1min. Moreover, the nitrate adsorption was governed by both pseudo first and second order kinetic model.

6.3 Biochar based nanocomposites:

Despite that BC is an efficient sorbent but many authors found that pristine biochar have low adsorption capacity (Wang *et al.*, 2015a) and its removal efficiency for arsenic is restricted for the following reasons: (i) predominance of negative charge on biochar restrict the sorption of negatively charged anions such as arsenite and arsenate (Mukherjee *et al.*, 2011; Yao *et al.*, 2012); (ii) Raw biochar hard to selectively adsorb pollutants; (Reigstad, #51) Biochar also increase the mobility and bioavailability of arsenic (Namgay *et al.*, 2010; Beesley *et al.*, 2014); (iv) pristine BC has a low desorbability potential for sorbed ions (Mosa *et al.*, 2020); (v) Powdered biochar requires centrifugation and filtration step which otherwise cannot be separated from aqueous medium (Yi *et al.*, 2019; Zhang *et al.*, 2019). Iron based nanoparticles may overcome all these limitations as they have magnetic properties. Here magnetic biochar is synthesized by Fe_3O_4 (Shan *et al.*, 2016), Fe_2O_3 (Zhu *et al.*, 2014), magnetic medium Fe (Li *et al.*, 2017) and biochar (Qin *et al.*, 2017) via either chemical co-precipitation or pyrolysis. Moreover, iron modified adsorbent are one of the most effective adsorbent for an extensive range of metals and metalloids including arsenic (Hao *et al.*, 2018; Hlongwane *et al.*, 2019) and can increase specific surface area. The adsorption of arsenite and arsenate on various biochar and iron oxide nanoparticles was studied. As expected Zhang *et al.* (2013) reported the adsorption of arsenic on novel magnetic biochar $\gamma\text{-Fe}_2\text{O}_3$ composite. These magnetic biochar composite showed a wonderful ferromagnetic properties having a saturation magnetization of 69.2 emu/g . Moreover, Langmuir maximum adsorption capacity of that novel magnetic biochar $\gamma\text{-Fe}_2\text{O}_3$ composite to arsenic is 3147 mg/kg . Similarly, Wang *et al.* (2015b) produced a magnetic biochar by pyrolysing pinewood along with natural hematite. Also in this Langmuir maximum adsorption capacity of arsenic for hematite pinewood biochar was doubled (429 mg/kg) then that of unmodified biochar (265 mg/kg). In a previous work (Khan *et al.*, 2020), corn straw derived biochar was used to support iron oxide nanoparticles for efficient removal of arsenic. The engineered magnetic biochar composites recorded the sorption capacity of 22.9 mgg^{-1} for arsenic based on Langmuir model. Similarly, Navarathna *et al.* (2019) observed the adsorption of (As III) on magnetic Douglas fir biochar composite which were prepared with a 29.2% wt of Fe_3O_4 loading

In a study by Zhang *et al.* (2016), As(V) removal from water by magnetic water hyacinth biochar was tested. The maximum sorption capacity As(V) was 7.41 mgg^{-1} and they also noted that small amount As(V) was reduced to As (Reigstad, #51) by magnetic water hyacinth biochar. This small conversion could possibly due to masking of reducing

functional groups of As(V) by impregnation of water hyacinth biochar with Fe-oxide layer. Bakshi *et al.* (2018) studied the retention of arsenic by a zero-valent iron (ZVI) biochar complex. This zero-valent composite recorded the good sorption capacity of As(V) over the range of contaminated drinking water pH(7-7.5). Moreover, this biochar composite could also promote the reduction of As(V) to As (Reigstad, #51) and oxidation of Fe^0 to Fe^3 and the co-precipitation of As^3 and Fe^3 leading to the formation of Fe(As)OOH onto biochar surfaces.

The sorption of fluoride on to magnetic biochar was examined by Dewage *et al.* (2018) using batch operational mode. The batch sorption data at optimal solution pH value 7 were described by Langmuir and Freundlich isotherm model with the sorption capacity of 9 mgg^{-1} for fluoride. Mohan *et al.* (2014) synthesized and used both magnetic and nonmagnetic corn stover biochar. In this batch sorption data at optimal solution pH value 2 were described by Redlich-Peterson isotherm model with the sorption capacity of 6.42 mgg^{-1} for fluoride. These magnetic biochar were further characterized by SEM, TEM, FTIR, EDXRF and Raman analyses. Moreover, in a study by Zhou *et al.* (2019), the biomass of bovine bone and peanut hulls was used to synthesize magnetic bone biochar to adsorb fluoride ions by co-precipitation. This magnetic bone biochar have an adsorption capacity of 5.23 for fluoride at pH 8. However, the sorption capacity of this magnetic biochar was reduced as the pH increases. This can be explained by the progressive deprotonation of the surface acidic functional groups which allow negative charge to increase and electrostatic repulsions

Biochar has been proven as the ideal adsorbent for the removal of hexavalent chromium ions from the aqueous environment, due to its economical, eco-friendly and high efficiency nature. However, the efficiency of pristine biochar is very low due to net negative surface charge (Li *et al.*, 2019) and provide only limited capacity to adsorb hexavalent chromium ions (Liu *et al.*, 2019). Additionally, the powdered biochar are not easy to separate from wastewater commonly require centrifugation and filtration step which limit the large scale application of biochar for wastewater treatment (Essandoh *et al.*, 2017; Yi *et al.*, 2019; Zhang *et al.*, 2019). Hence, in order to change negatively charged surface and improve the separation and recovery of biochar, magnetic particles (Fe^0 , $\gamma\text{-Fe}_2\text{O}_3$ and Fe_3O_4 , etc) have recently been introduced into biochar matrices to enhance its capacity for Cr(VI) removal (Liang *et al.*, 2019; Yi *et al.*, 2020). Several modification methods have been developed to form magnetic biochar to enhance their adsorption of Cr(VI). For example, The adsorption performance of Cr(VI) using Fe_3O_4 modified biochar was studied by (Liang *et al.*, 2019), reporting the adsorption capacity of 55 mgg^{-1} at pH 2. They justified that carbon skeleton of biochar provide binding and reducing sites for Cr(VI) ions, while newly formed Cr (Reigstad, #51) ions were mainly immobilized by Fe_3O_4 nanoparticles, thus achieving a ultra-high adsorption of chromium ions. Another

similar research was conducted by Zhong *et al.* (2018), who synthesized magnetic biochar with active Fe_3O_4 nanoparticles to remove Cr(VI) ions from water. They claimed that Cr(VI) removal efficiency of the magnetic biochar (84.3%) was higher than the pristine biochar (26.5%). They suggested that the excellent adsorption capacity of magnetic biochar was due to Fe_3O_4 which increase the pore volume, BET-surface area which increase the adsorption of Cr(VI) ions. These Fe_3O_4 nanoparticles also produced active carbon- centered persistent free radicals and development of graphite structure to enhance the Cr(VI) reduction. Furthermore, magnetic biochar synthesized by co-pyrolysis of nano-zero-valent iron and sewage sludge has the removal capacity of Cr(VI) was 11.56 mgg^{-1} at pH 2 (Liu *et al.*, 2020). The key mechanisms involving remediation of Cr(VI) from different environmental matrices by biochar include precipitation, electrostatic attraction, chemisorption, complex formation and reduction of Cr(VI) to Cr (Reigstad, #51) (Liu *et al.*, 2022).

Zhang, X. *et al.* (2018) prepared magnetic biochar derived from *Melia azedarach* wood for adsorption of Cr(VI) from water, reporting the adsorption capacity of 25.27 mgg^{-1} . The composite was able to remove 99.8% Cr(VI) and the maximum adsorption occurred at pH 3 and Cr(VI) concentration of 10 mg/L . Furthermore, removal of Cr(VI) followed adsorption-reduction-adsorption process following Langmuir and pseudo-second-order kinetic model. Similar study was conducted by Yang *et al.* (2017) in removing Cr(VI) using magnetic biochar. They reported that the maximum adsorption capacity was 42.7 mgg^{-1} at 30°C and pH 2 through 180min. The authors found that the experimental data fitted well with pseudo-second-order model for kinetics and Langmuir model for isotherm with correlation $R^2=0.9996$ and $R^2>0.9980$, respectively. Further, Freundlich and Evolich model in this research indicating that there is an existence of chemisorption during adsorption process which forces Cr(VI) ions to porous structure. In another study by Mahanty and Mondal (2021), the agricultural waste magnetic biochar was used to remove Cr(VI) ions from aqueous solution. The results exhibited that the magnetic biochar had achieved a remarkably high adsorption of 75.66 mgg^{-1} at pH 2. These authors also claimed that the magnetic biochar have high Cr(VI) removal efficiency (95.27%) then the pristine biochar (45.56%). Moreover, the presence of coexisting cations had a slight interference on the adsorption of Cr(VI) on magnetic biochar. Also the interference of coexisting cations depends on solubility of electrolytes and size of cations and follows the order $\text{Al}^{3+} < \text{Mg}^{2+} < \text{Na}^+$. Magnetic biochar derived from burely tobacco stem was successfully applied by Cui *et al.* (2022) as a novel and effective adsorbent material for the removal of Cr(VI) from waste water. The authors highlighted that the adsorption of Cr(VI) was followed by three steps: Cr(VI) ions were reduced to Cr (Reigstad, #51) ions, after that these ions were adsorbed and exchange hydroxyl ions

from magnetic biochar then, amino acids groups of magnetic biochar form complex with Cr (Reigstad, #51) ions. The maximum adsorption capacity of Cr(VI) calculated from Langmuir model was 58.74 mgg^{-1} , which indicate potential maximum adsorption ability. Santhosh *et al.* (2020) reported that the incorporation of magnetic particles into raw biochar through co-precipitation had increased the BET surface area. They also observed that adsorption data fitted well with Langmuir model with maximum sorption capacity of 80.96 mgg^{-1} at pH 2. They also claimed that desorption of Cr(VI) ions increases at high pH, since CrO_4^{2-} is the dominant form of Cr(VI) in alkaline solution which can be exchange by hydroxyl anions. This desorption of ions showed that this adsorbent could be re-used successfully for Cr(VI) adsorption. Ma *et al.* (2019) prepared magnetic biochar from cotton stalk and they fitted the adsorption data of Cr(VI) into Sips isotherm model. The also reported that the saturation magnetization value of magnetic corn stalk biochar was 27.59 emu/g . Batch adsorption experiment results indicated that maximum adsorption capacity for magnetic biochar was 20.05 mgg^{-1} which was higher than that of pristine biochar (18.77 mgg^{-1}). A list of magnetic nanoparticles with their adsorption capacities are summarized in **Table 3**.

Recent development, include the successful removal of phosphate by magnetic biochar derived from bamboo biomass (Zhu, Z. *et al.*, 2018). The field emission scanning electron microscope (FE-SEM) pattern shows the presence of three distant pores (micro, meso and macropores with a total pore volume of $0.12 \text{ cm}^3/\text{g}$). Also experiment was investigation by changing different parameters with the inferences. (a) Adsorption efficiency was reduced by increasing the temperature. (b) Also, by increasing the initial phosphorus concentration leads to higher adsorption of phosphate. Hence, Cai *et al.* (2017) investigated the adsorption capacity of phosphate on to magnetic hyacinth biochar. It was found that this magnetic hyacinth biochar reached $Q_{\text{max}} = 5.07 \text{ mgg}^{-1}$ at pH 5 and 25°C . Moreover, the adsorbent selectivity of magnetic hyacinth biochar was scrutinized in the presence of wide selectivity of ions including arsenate, carbonate, nitrate, sulfate and antimonate. Significant decline in adsorption capacity of phosphate was observed in the presence of arsenate and carbonate while the inhibition effect of other ions are less significant. In attempt Karunanayake *et al.* (2019) also scrutinized the adsorption removal of phosphate ions by using magnetic douglas fir biochar. It was found that maximum adsorption capacity of phosphate onto magnetic douglas fir biochar was 91.3 mgg^{-1} at pH 3 and 45°C . Moreover, Fe-OH group foster adsorption of phosphate ions by electrostatic interaction, H-bonding and dipole-dipole interaction. In yet another attempt, He *et al.* (2021) reported highly efficient magnetic biochar for the removal of phosphate from water. These composites revealed an efficient adsorption behavior towards phosphate ions at which q_{max} was 8.93 mgg^{-1} at $\text{pH} < 8$ with a adsorbent dosage of $0.5\text{-}20 \text{ mg/L}$. Moreover, the plausible adsorption

mechanism of phosphate onto magnetic biochar composite was fully explained by the spectral techniques and impact of pH, suggesting the hydrogen bonding and electrostatic interaction. In another study, Zhang and his co-workers reported the removal of phosphate from aqueous solution by nanoporous biochar iron oxides (Zhang, Z. *et al.*, 2020). They found that the maximum adsorption capacity was 9.408, 13.81, 18.49 and 22.14 mgg^{-1} phosphate at pH 2 for magnetite, hematite, ferrihydrite and goethite biochar, respectively. Moreover, the order was consistent with the point of zero charge (PZC) of the magnetic biochar, in which magnetite has lowest PZC and the goethite has highest PZC. Besides, the adsorption process was well-fitted by the pseudo-second order kinetic model indicating a chemisorption process. Another, porous magnetic biochar was inspected as an adsorbent for the efficient removal of phosphate from water (Tomin and Yazdani, 2022). It was found that this magnetic porous biochar exhibited complete removal of phosphate (100%) at the lower concentration (5 mg/l P) due to its highest specific surface area ($93 \text{ m}^2/\text{g}$). Furthermore, the adsorption process was well-fitted with Langmuir isotherm and pseudo-second order kinetic model. Oprescu *et al.* (2022) studied the sorption of nitrate on magnetic biochar derived from corn stalk prepared by pyrolysis process. Various factors affecting the removal of nitrate including contact time, pH, mass of adsorbent and nitrate concentration were also studied. It was found that this magnetic biochar showed higher removal efficiency of nitrate (98.91%) at pH 7, 0.25 g adsorbent, 50 ppm of nitrate concentration and 120 min . In one investigation Wei *et al.* (2018) evaluated biochar supported nano zerovalent-iron composite as a propitious adsorbent for nitrate removal from ground water. Also, preferable nitrate removal (75%-97%) was obtained over a wide range of pH (2-12). They also concluded that biochar favor nitrate reduction by directly mediating redox potential, electron transfer and pH

6.4 Magnetic-zeolite nanocomposite:

Natural zeolite have attracted much attention in recent years for the removal of pollutants from wastewater due to its high adsorption capacity and efficiency, high ion exchange capacity, low cost, high specific surface area, high chemical stability and abundance (Gaffer *et al.*, 2017). Due to their superior properties, zeolite have been used for the removal of various anions pollutants (Hrenovic *et al.*, 2008; Yusof and Malek, 2009). However, centrifugation and filtration techniques have been required to recover this zeolite after the adsorption process. Various methods have been used to improve the adsorption capacity of zeolite. One method to improve the adsorption capacity of natural zeolite is through transforming into magnetic material (Asanu *et al.*, 2022). One of the most investigated utilization concerns As removal from wastewater, owing to the high selectivity of zeolite. The removal of As(V) was carried out by zeolite functionalized with nanoscale zero-valent iron (Suazo-Hernández *et al.*, 2019). In a batch mode, the highest capacity of magnetic

Table 3 Novel nano-magnetic biochar applied for the removal of anions from water

Anions species removal	Feedstock	Reagent	Isotherm-model	Kinetic model	pH	Adsorption capacity (mg/g)	References
As(V)	Cottonwood	$\gamma\text{-Fe}_2\text{O}_3$	Langmuir	Pseudo first-order model	-	3147	(Zhang <i>et al.</i> , 2013)
As III	Douglas fir	Fe_3O_4	The Sips isotherm	Pseudo second order model	- 7	5.49	(Navarathna <i>et al.</i> , 2019)
As III	Corn straw	Fe_3O_4	Langmuir	Pseudo second order model	- 4	22.94	(Khan <i>et al.</i> , 2020)
Cr(VI)	Cotton stalk	Fe_3O_4	The Sips isotherm	Pseudo second order model	- 2	20.05	(Ma <i>et al.</i> , 2019)
Cr(VI)	Rice husk	Fe_3O_4	-	-	-	8.35	(Zhong <i>et al.</i> , 2018)
Cr(VI)	Sewage sludge	Fe_3O_4	Langmuir	Pseudo second order model	- 2	11.56	(Liu <i>et al.</i> , 2020)
Cr(VI)	Wood chip	Fe_3O_4	Langmuir	Pseudo second order model	- 2	80.96	(Santhosh <i>et al.</i> , 2020)
Cr(VI)	Peanut husk	$\gamma\text{-Fe}_2\text{O}_3$	Freundlich	Pseudo second order model	- 2	8.51	(Mahanty and Mondal, 2021)
F^-	Douglas fir	$\alpha\text{-Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4$	Langmuir	-	7	9.04	(Dewage <i>et al.</i> , 2018)
F^-	Corn stover	$\text{Fe}^{3+}/\text{Fe}^{2+}$	Langmuir	Pseudo first-order model	- 2	4.11	(Mohan <i>et al.</i> , 2014)
F^-	Bone derived biochar	$\gamma\text{-Fe}_2\text{O}_3$	Langmuir	-	8	5.23	(Zhou <i>et al.</i> , 2019)
NO_3^-	Corn stalk	Fe_3O_4	-	-	7	19.67	(Oprescu <i>et al.</i> , 2022)
P^{-1}	Douglas fir	Fe_3O_4	Langmuir	-	3	91.3	(Karunanayake <i>et al.</i> , 2019)
P^{-1}	Tobacco stalk biochar	Fe_3O_4	Langmuir	Pseudo second order model	- <8	8.93	(He <i>et al.</i> , 2021)
P^{-1}	Coconut shell	Fe_3O_4 $\text{Fe}(\text{OH})_3$ $\alpha\text{-FeOOH}$ $\alpha\text{-Fe}_2\text{O}_3$	Langmuir	Pseudo second order model	-	9.408 18.49 22.14 13.81	(Zhang, Z. <i>et al.</i> , 2020)
P^{-1}	Spruce sawdust	Fe_2O_3	Langmuir	Pseudo second order model	-	3.47	(Tomin and Yazdani, 2022)

zeolite for As(V) was found to be 47.30 mgg^{-1} . The results showed that removal process of As(V) had well followed

Pseudo-second-order model. Saleem Attia and colleagues have contributed extensively to the development and application of magnetic zeolite composite for As contaminated wastewater. The authors thoroughly characterized this magnetic zeolite using TEM, EDX, FTIR and XRD techniques. The results obtained demonstrated that the removal percentage of As could reach over 95.6% at a pH value of 2.5. The authors emphasized that the magnetic zeolite composite exhibited high practical applicability as the As adsorption efficiency had retained upto 93.95% after 5 regeneration cycle (Saleem Attia *et al.*, 2014).

Asanu *et al.* (2022) Prepared a zeolite coated with magnetic nanoparticle for Cr (VI) removal from aqueous solution. Here, high Cr (VI) removal efficiency (93.57%) was observed at adsorbent dosage of 2g/L, contact time 75min and at pH 1.5. They observed that experimental data fitted well to the Langmuir isotherm model with a determination coefficient of $R^2=0.9966$, implying the presence of monolayer. Liu and Cao (2019) used magnetic zeolite composite for the removal of F^- from water. They observed that the adsorption of F^- decreases by increasing the concentration of iron content. In another report magnetic zeolite composite was synthesized and employed for the removal of phosphate from water (Xu *et al.*, 2020). A good adsorption capacity of 38.91 mgg^{-1} was observed under optimum condition. Besides, the adsorption process was well-fitted by the pseudo-second order kinetic model indicating a chemisorption process

6.5 Magnetic-polyaniline (PANI):

Polymers are a class of great interest because of their high surface area, excellent mechanical property, adjustable surface topography, high surface area, desired pore structure and economical regeneration under mild conditions (Zare *et al.*, 2018). Among various polymers, polyaniline (PANI) has received considerable interest to remove aqueous pollutants due to its versatile functional groups, tunable morphological properties and simple synthesis procedure (Maponya *et al.*, 2019). Another advantage associated with this PANI-based nanocomposites is their morphological diversity which further adjusted by changing some synthesis parameters such temperature, dopant type and reaction time. Consequently, various aqueous pollutants were removed by fabrication of this PANI-based nanocomposites (Samadi *et al.*, 2021). Bhaumik and coworkers have reported the removal of arsenic from aqueous by polyaniline/ Fe° composite. The adsorption was analyzed with pseudo first order and pseudo-second-order kinetic model. The pseudo-second-order was found to be a best-fitted model. The adsorption of As(V) and As(Reigstad, #51) followed the Langmuir isotherm model with the maximum adsorption capacity 227.3 and 232.5mg/L at pH=7. They also observed that there had no significant effect of coexisting anions (HCO_3^- , SO_4^{2-} , SiO_3^{2-}) on the

removal of As(V) and As(Reigstad, #51) (Bhaumik *et al.*, 2015). Prepared core-shell structured magnetic nanocomposite for enhanced removal of As(V) adsorption. The adsorbent was characterized by FTIR, XRD, SEM, XPS, TGA and BET methods. Moreover, adsorption process followed a pseudo-second-order and Redlich- Peterson isotherm model indicating that physical and chemical adsorption taking place on the homogenous and heterogeneous adsorption interface (Zhang, Y. *et al.*, 2020). Zhou *et al.* (2015) explore the application of magnetic-core shell nanoparticles Fe_2O_3 polyaniline composite for the removal of As(V) and As(Reigstad, #51) from water. The maximum adsorption capacity of As(V) and As(Reigstad, #51) were 1.385 and 1.066 mg/L, respectively. Lyu *et al.* (2019) prepared coral-like polyaniline magnetic adsorbent and then used it in batch removal of Cr(VI) from aqueous solution. In this study effect of temperature, adsorbent dosage, pH of the solution on the adsorption of Cr(VI) over magnetic polyaniline were studied. It was observed that magnetic polyaniline exhibited excellent adsorption over magnetic polyaniline in acidic range of pH and lower temperature. Moreover, Cr(VI) removal efficiency was not changed significantly in the presence of competing ions i.e., Cl^- , NO_3^- , SO_4^{2-} and H_2PO_4^- . In a similar type of studies Gu *et al.* (2012) prepared magnetic polyaniline nanocomposite (PNC) for the adsorption of Cr(VI). Preliminary investigation ascertained the optimum adsorption at pH=7, Cr(VI) concentration 1-3mg/L, 10mg amount of adsorbent and 5min of contact time at room temperature. Ebrahim *et al.* (2016) also investigated the removal of noxious Cr(VI) from aqueous environment by using akaganeite polyaniline composite. This composite were initially characterized by FTIR, SEM, XRD and HRTEM. Polyaniline/ Fe_3O_4 nanocomposite were synthesized by Jokar *et al.* (2014) and their suitability as fluoride was examined. The maximum adsorption capacity (85%) was achieved at the adsorbent dosage of 0.25g of polyaniline/ Fe_3O_4 nanocomposite. The authors claimed that by increasing the amount of adsorbent dosage significantly influenced the adsorption of fluoride ions due to increase in number of active sites

7. Conclusion:

It is evident from the above thorough and critical discussion that the anions pollution in water is a global issue, and various treatment methods have been developed for the removal of these anionic pollutants from water sources. Iron oxide nanoparticles have great potential for the removal of these anions due to its unique physical and chemical properties. The present review article primarily emphasized the recent advancements for the use of iron oxide nanoparticles and their composite as efficient and cost-effective adsorbent for removal of anions from water. Iron oxide nanoparticles and their composites having variable morphologies, variable surface chemistry, distinct crystalline nature and tunable band edges, possess excellent ability to remediate anion pollutants from different water sources,

based on various adsorptive pathways i.e., precipitation, chemisorption, physisorption, ion exchange, and so forth. The adsorption of a wide range of anions based on As(III), As(V), Cr(VI), F^- , NO_3^- , P^{3-} over a plethora of iron oxide-based adsorbents like hematite, goethite, magnetite, akaganeite, lepidocrocite, wustite, and their composites viz graphene/grapheneoxide/reduced graphene oxide, biochar and chitosan are critically reviewed

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8. Conflict of Interest

The authors declare no conflict of interest.

9. Authors' Contribution

Aimen Ali, Hamna Bashir: Investigations, Data Curation and Visualization, Writing Original Draft; Irshad Bibi, Nabeel Kahn Niazi: Supervision, Writing reviews and corrections.

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