

# Synergetic Effect of Activated Carbon and Zero-valent Iron for Removal of Nitrate-polluted Potable Water

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**Abstract:** Nitrate contamination in drinking water poses significant health risks, necessitating effective remediation techniques. This study investigates the synergetic effect of combining activated carbon (AC) and nano-zero valent iron (nZVI) for the removal of nitrate ions from polluted potable water. nZVI was synthesized via the reduction of ferric ions with sodium borohydride and characterized using SEM, TEM, EDX, and XRD analyses, revealing spherical nanoparticles with an average size of 40 nm and high crystallinity. AC was prepared through phosphoric acid activation and characterized similarly. Batch experiments were conducted to evaluate the nitrate removal performance of nZVI and the nZVI-AC composite under varying conditions. Results showed that nZVI effectively reduced nitrate concentrations, with optimal dosages achieving up to 96% removal. The nZVI-AC composite further enhanced the removal efficiency, minimizing the formation of ammonia as a by-product. The adsorption capacity of AC for nitrate ions was also investigated, demonstrating its complementary role in the treatment process. The synergetic effect of nZVI and AC offers a promising approach for efficient nitrate remediation in drinking water, with potential applications in water purification and environmental remediation.

**Keywords:** Nitrate removal; Drinking water treatment; Nano-zero valent iron (nZVI); Activated carbon; Adsorption; Water purification..

## 1. Introduction

Nitrate contamination in groundwater and drinking water supplies has emerged as a major environmental and public health concern globally. Nitrate ( $\text{NO}_3^-$ ) is a water-soluble anion that is part of the natural nitrogen cycle, involving the interchange of nitrogen between the atmosphere, land, and living organisms [1]. However, excessive nitrate levels in potable water can have severe health implications, particularly for infants and pregnant women.

The presence of nitrate ions in drinking water above permissible limits is considered a significant environmental problem that can fatally affect human health [2]. The World Health Organization (WHO) has set the maximum acceptable concentration of nitrate in drinking water at 50 mg/L, while the United States Environmental Protection Agency (USEPA) has established a limit of 45 mg/L [3]. Nitrates can enter groundwater sources through various pathways, including agricultural runoff from excessive use of nitrogenous fertilizers, industrial processes, animal waste decomposition, and the oxidation of ammonia-containing wastewater [4, 5].

Nitrate is one of the most stable forms of nitrogen oxidation states, making it difficult to reduce and remove from water sources [2]. Although nitrate is chemically inert, it can be degraded through microbial processes or chemical reduction techniques. Various methods have been employed for the

removal of nitrate from aqueous solutions, including electro dialysis, reverse osmosis, catalysis, electrocatalytic reduction, membrane bioreactors, ion exchange, and biological denitrification processes [6].

Ion exchange and reverse osmosis are widely used techniques for nitrate removal, but they have limitations, such as the generation of concentrated brine streams and high operational costs [7, 8]. Biological denitrification processes, while effective, require strict control over environmental conditions and can be susceptible to microbial contamination [9].

Chemical denitrification, involving the reduction of nitrate using various chemical-reducing agents, has emerged as a promising alternative. In this context, nano-zero-valent iron (nZVI) has garnered significant attention as an efficient and environmentally friendly material for groundwater remediation [3, 10]. nZVI possesses unique properties, including small particle size, large specific surface area, strong reducibility, and high surface activity, making it highly effective in degrading pollutants and reducing nitrate ions [11].

Additionally, activated carbon (AC) adsorbents have demonstrated high efficiency in adsorbing nitrate ions, with relatively low equilibrium contact times and high adsorption levels [12]. The combination of nZVI and AC could potentially exploit their synergetic effects, leading to enhanced nitrate

removal and minimizing the formation of undesirable by-products, such as ammonia.

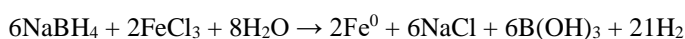
Various methods have been employed for the removal of nitrate from aqueous solutions, including electro dialysis, reverse osmosis, ion exchange, and biological denitrification. However, each of these techniques has inherent limitations. Electro dialysis and reverse osmosis, while effective, often generate concentrated brine streams that pose disposal challenges and involve high operational costs. Ion exchange is efficient but can be limited by the need for frequent resin regeneration and the risk of secondary pollution. Biological denitrification processes require strict control over environmental conditions and are susceptible to microbial contamination, making them less reliable. Chemical reduction methods, such as the use of nanosized zero-valent iron (nZVI), have shown promise due to their simplicity and effectiveness. However, conventional nZVI can lead to incomplete reduction and the formation of undesirable by-products like ammonia. In this study, we propose a novel approach combining nano-zero valent iron (nZVI) with activated carbon (AC) to exploit their synergistic effects, thereby enhancing nitrate removal efficiency while minimizing by-product formation. This innovative combination addresses the limitations of existing methods and provides a more effective solution for nitrate remediation in drinking water.

## 2. Materials and methods

**Materials** The key materials used in this study included sodium borohydride, absolute ethanol, ferric chloride hexahydrate (from El-Nasr company), potassium nitrate, phosphoric acid (from Merck), activated carbon (from El-Nasr company), and aniline monomer (from Sigma-Aldrich). All chemicals were used as received without further purification. Deionized (DI) water was employed for solution preparation and rinsing purposes.

### 2.1. Synthesis of Nano-Zero Valent Iron (nZVI)

A nZVI was optimized by varying the molar ratio of sodium borohydride to ferric chloride, reaction temperature, and stirring speed to achieve uniform particle size and high reactivity. The optimal conditions were found to be a 0.1 M NaBH<sub>4</sub> solution added dropwise to a 0.1 M FeCl<sub>3</sub> solution at room temperature with constant stirring, resulting in nZVI particles with an average size of 40 nm and high crystallinity. The reaction as follows:



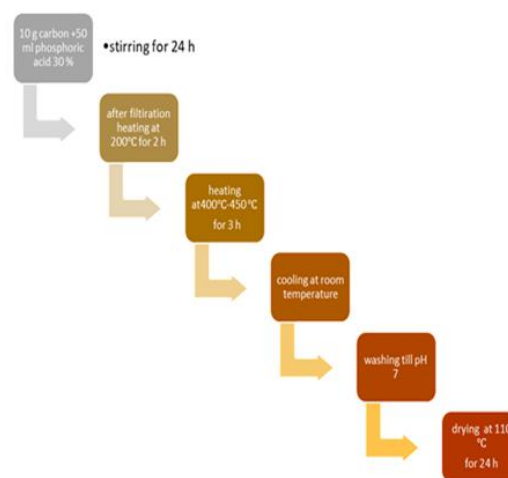
### 2.2. Synthesis of Activated Carbon (AC)

For the preparation of AC, the optimization focused on the impregnation ratio of phosphoric acid to carbon precursor, carbonization temperature, and time. The optimal conditions were a 1:1 impregnation ratio, carbonization at 450°C for 3 hours, and thorough washing to neutral pH, producing AC with a high surface area and effective pore structure for adsorption [13, 14].

### 2.3. Characterization Techniques:

Nano-Zero Valent Iron (nZVI) Characterization, the synthesized nZVI was characterized using various techniques. Scanning electron microscopy (SEM) using a JEOL JSM 6510 lv instrument examined surface morphology and particle size. Energy dispersive X-ray spectroscopy (EDX) with an Oxford X-Max 20 system analyzed elemental composition. Transmission electron microscopy (TEM) using a JEOL JEM-2100F provided high-resolution imaging for morphology and size distribution. X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer with Cu K $\alpha$  radiation investigated crystalline structure, phase composition, and average crystallite size (via Scherrer equation).

For activated carbon, SEM using a JEOL JSM-IT200 examined surface morphology and pore structure before and after activation. EDX analyzed elemental composition. XRD using the same Bruker diffractometer studied crystalline structure and phase composition.



**Fig. 1:** Preparation of activated carbon

### 2.4. Nitrate Removal Experiments

Nitrate Removal by nZVI Batch experiments evaluated nZVI performance in nitrate removal from aqueous solutions. nZVI dosages were added to nitrate solutions of known concentrations and mechanically stirred using a jar test apparatus. Reaction conditions like contact time and initial nitrate level were varied. Residual nitrate was measured using a Hach DR 6000 spectrophotometer for synthetic and naturally contaminated water.

The synergistic effect of nZVI and AC was studied by batch experiments using their composite for nitrate removal under varied conditions like dosage and contact time. Residual nitrate was measured spectrometrically. AC's nitrate adsorption was also examined alone by batch tests varying initial nitrate levels and using a fixed AC dose with mechanical stirring and residual nitrate measurement.

## 3. Results and Discussion

### 3.1. Zero Valent Iron Nanoparticles Characterization

#### 3.1.1. SEM, TEM, and XRD Characterization

According to the SEM study, the nZVI particles produced under the optimized conditions exhibit a spherical morphology and appear to be agglomerated or clustered together as shown in Figure 1 a. The majority of the prepared nZVI particles are within the nanoscale range, with an average particle size of approximately 40 nm. It is noted that the particles are relatively homogeneous in size and shape, and possess a uniform shade or contrast in the SEM micrographs.

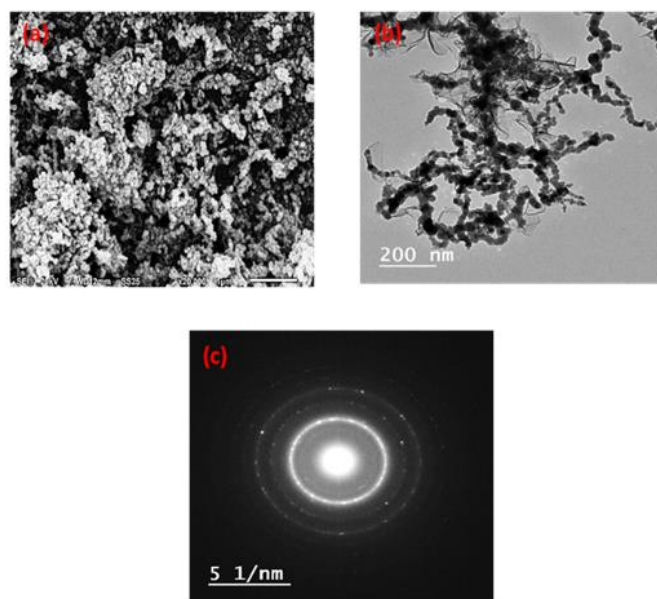
The energy dispersive X-ray analysis (EDX) of the prepared nZVI nanoparticles shows a strong signal corresponding to the iron region, confirming the successful generation of iron-based nanoparticles with a high percentage or content. A relatively minor signal attributed to oxygen is also observed at around 0.5 keV. This oxygen signal may result from the partial oxidation of the highly reactive nZVI particles upon exposure to air during sample transfer and analysis.

The transmission electron microscopy (TEM) analysis provides a detailed description and characterization of the morphology and size distribution of the prepared nZVI as depicted in **Fig. 1b**. The recorded TEM micrographs reveal that the nanoparticles exhibit a cluster-like or agglomerated morphology, with the individual nanoparticles having sizes ranging from approximately 13 nm to 40 nm. The nanoparticles appear to be interconnected, forming branched structures or chains.

The selected area electron diffraction (SAED) patterns obtained from the TEM analysis further confirm the crystalline nature of the synthesized nZVI nanoparticles as shown in Figure 1 c. The SAED patterns shown in the file (Figure 1c) exhibit distinct diffraction rings or concentric circles, which are characteristic of polycrystalline or nanocrystalline materials. The presence of these diffraction rings indicates that the synthesized nZVI nanoparticles are crystalline in nature and consist of multiple crystalline domains or grains with different orientations. The presence of diffraction rings corresponding to the (110), (200), (211), and (220) planes is consistent with the expected BCC crystal structure of zero-valent iron nanoparticles.

The SEM and TEM characterization results provide valuable insights into the morphological, structural, and size characteristics of the synthesized nZVI nanoparticles. The spherical morphology and nanoscale size range observed in the SEM and TEM analyses are desirable features for nZVI, as they contribute to a high surface area-to-volume ratio, which can enhance the reactivity and adsorption properties of the nanoparticles. The agglomeration or clustering behavior observed in both SEM and TEM micrographs is a common phenomenon for nZVI due to the high surface energy of the nanoparticles and their tendency to minimize surface area. While agglomeration can potentially reduce the available surface area, the interconnected nanoparticle clusters may still provide accessible active sites for reactions and adsorption processes. Overall, the SEM and TEM characterization results confirm the successful synthesis of nZVI nanoparticles with the desired morphological and structural characteristics, making them suitable candidates for applications in

environmental remediation, such as nitrate removal from water.



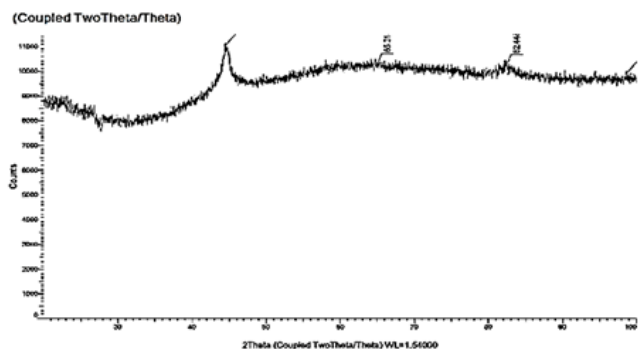
**Fig. 1:** SEM micrographs (a) TEM (b), and SAED analysis (c) of the prepared nZVI nanoparticles at optimum conditions.

The XRD pattern of the synthesized nZVI, as shown in **Fig. 2**, exhibits four distinct diffraction peaks or signals at  $2\theta$  angles of  $44.67^\circ$ ,  $65.018^\circ$ ,  $82.327^\circ$ , and  $98.94^\circ$ . These sharp and well-defined diffraction peaks indicate the presence of crystalline phases within the nZVI sample. The observed diffraction peaks correspond to the crystalline zero-valent iron (Fe0) lattice with a cubic structure. The specific Miller indices assigned to these peaks are (110), (200), (211), and (220), respectively which are in agreement with that obtained from SAED.

The presence of these diffraction peaks at the expected  $2\theta$  positions for a body-centered cubic (BCC) iron crystal structure confirms the successful synthesis of zero-valent iron nanoparticles. The BCC structure is the characteristic crystal structure of metallic iron (Fe0) at room temperature. The sharpness and intensity of the diffraction peaks suggest that the synthesized nZVI nanoparticles possess a high degree of crystallinity and long-range atomic ordering within the iron lattice. Sharp diffraction peaks are typically observed for well-crystallized materials, while broad or diffuse peaks are indicative of amorphous or poorly crystalline structures.

The average crystallite size of the nZVI nanoparticles was estimated from the XRD data using the Scherrer equation. The Scherrer equation relates the peak broadening or full width at half maximum (FWHM) of the diffraction peaks to the average crystallite size, taking into account factors such as the X-ray wavelength and diffraction angle [15-17]. The calculated average crystallite size for the synthesized nZVI nanoparticles is reported to be 12.23 nm. This value is consistent with the particle size range observed in the electron microscopy (SEM and TEM) analyses, which showed nanoparticles with sizes ranging from approximately 13 nm to 40 nm.

Overall, the XRD analysis provides valuable information about the crystalline structure, phase composition, and average crystallite size of the synthesized nZVI nanoparticles. The results confirm the successful synthesis of zero-valent iron nanoparticles with the desired BCC crystal structure and high crystallinity, which are essential for their intended applications in environmental remediation or catalytic processes.



**Fig. 2:** XRD pattern of nZVI synthesized at optimum conditions.

The SEM analysis was carried out to examine the surface morphology and pore structure of the activated carbon samples, both before and after the activation process. The SEM micrographs presented show a noticeable difference in the surface morphology and pore structure between the non-activated and activated carbon samples as shown in **Fig. 3**. The activated carbon sample exhibits a more porous and irregular surface compared to the non-activated carbon. The development of a porous structure is a characteristic feature of activated carbon materials. The activation process, which involves treatment with phosphoric acid and high-temperature carbonization, facilitates the creation of pores and increases the surface area of the carbon material. This porous structure is essential for enhancing the adsorption capacity and performance of activated carbon in various applications, including the removal of pollutants from water.

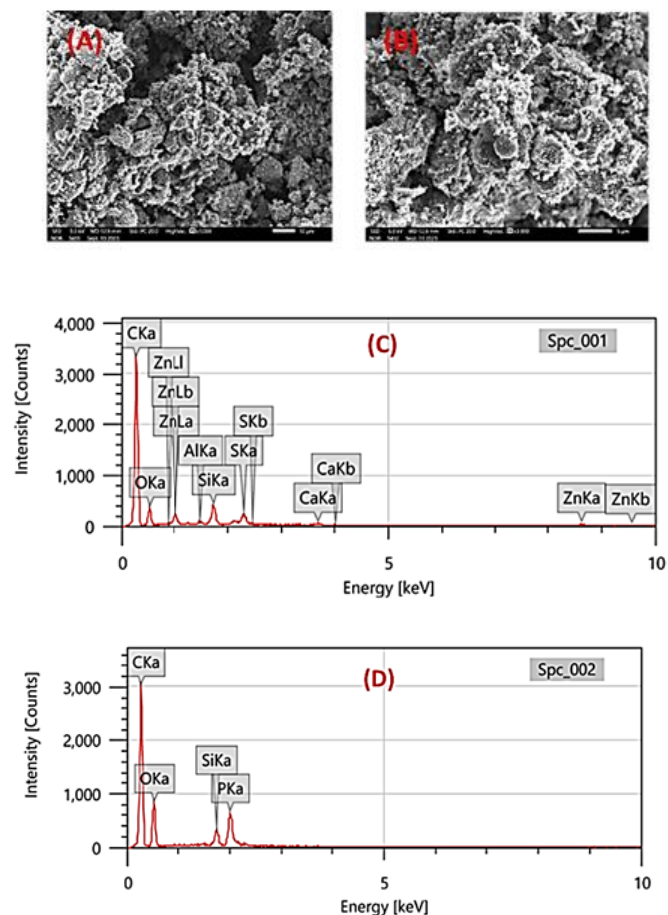
The elemental composition of the non- and activated carbon samples was analyzed using an EDX system attached to the SEM instrument as in **Figs. 3C and 3D**. The EDX spectra provided in the file show a difference in the elemental composition between the non-activated and activated carbon samples.

The EDX analysis allows for the identification of the elemental constituents present in the activated carbon samples. The observed differences in the elemental composition can be attributed to the activation process, which may involve the removal of certain elements or the incorporation of new elements from the activating agent (in this case, phosphoric acid).

The crystalline structure and phase composition of the activated carbon samples were analyzed using an X-ray diffractometer. The XRD patterns of the non-activated and activated carbon samples are presented in **Fig. 4**. The XRD patterns provide information about the crystalline or

amorphous nature of the carbon materials. Activated carbon typically exhibits a broad or diffuse XRD pattern, indicating a predominantly amorphous or disordered structure. However, the presence of any sharp diffraction peaks may indicate the existence of crystalline phases, which could be attributed to the presence of impurities or the formation of specific crystalline structures during the activation process. By comparing the XRD patterns of the non-activated and activated carbon samples, changes in the crystalline or amorphous nature of the material can be investigated. These changes may be related to the removal or incorporation of specific phases or the rearrangement of the carbon structure during the activation process.

Overall, the characterization techniques employed for the activated carbon samples provide valuable insights into the morphological, structural, and compositional changes induced by the activation process. These changes are directly related to the enhanced surface area, porosity, and adsorption properties of the activated carbon material, making it suitable for applications such as the removal of nitrate ions or other pollutants from water.



**Fig. 3:** SEM and EDX for the non-activated carbon (A, C) and activated carbon (B, D), respectively.

## 3.2. Nitrates Removal

### 3.2.1. Nitrates Removal by nZVI

**Fig. 5** shows the performance of the synthesized nZVI in removing nitrate ions from aqueous solutions. The experiment was conducted using an initial nitrate concentration of approximately 100 ppm, and the dosage of nZVI was varied from 0.5 to 3.0 g/L. The contact time for the experiment was maintained at 30 minutes at ambient temperature (25°C). The figure illustrates that the synthesized nZVI exhibited high efficiency in removing nitrate ions from the aqueous solution. As the dosage of nZVI increased, the removal efficiency also increased, indicating a positive correlation between the nZVI dosage and the extent of nitrate removal.

The high efficiency of the synthesized nZVI in removing nitrate ions can be attributed to several factors:

- Small particle size and high surface area: The small particle size (average of 40 nm) and spherical morphology of the nZVI result in a large surface area-to-volume ratio. This high surface area provides abundant active sites for the adsorption and reduction of nitrate ions.
- Highly crystalline structure: The XRD analysis confirmed the highly crystalline nature of the synthesized nZVI, which can enhance their reactivity and stability during the nitrate removal process.
- Reduction mechanism: nZVI are known to be effective reducing agents for various contaminants, including nitrate ions. The proposed mechanism involves the transfer of electrons from the nZVI to the nitrate ions, leading to their reduction and subsequent removal from the aqueous solution.

The observed increase in nitrate removal efficiency with increasing ZVI dosage can be attributed to the availability of more active sites and a higher overall surface area for the adsorption and reduction of nitrate ions. However, it is important to note that there may be an optimal dosage beyond which the removal efficiency plateaus or decreases due to factors such as particle agglomeration or saturation of active sites.

Overall, the results demonstrate the promising potential of the synthesized ZVI nanoparticles for the effective removal of nitrate ions from water sources. The combination of their small size, high surface area, and reducing capabilities makes them an attractive material for environmental remediation applications, particularly in the treatment of nitrate-contaminated water.

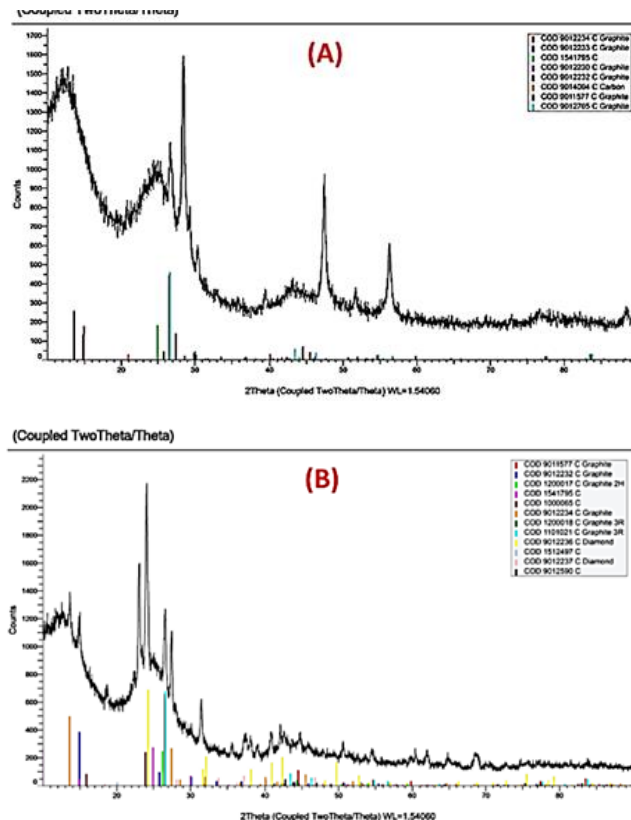
### 3.2.2. Nitrate removal by nZVI and activated carbon

**Fig. 6** (A and B) presents the nitrate removal and ammonia residual performance of the nZVI-AC composite. Figure 6A shows the effect of varying doses of activated carbon (0 - 3 g/200 mL) on the nitrate concentration after treatment, using a fixed dose of 1 g/L nZVI and an initial nitrate concentration of approximately 50 mg/L. The residual nitrate concentrations ranged between 34 mg/L and 42 mg/L, depending on the AC dose.

**Fig. 6B** illustrates the concentration of ammonia, a potential side product, resulting from the treatment process using the nZVI-AC composite. The ammonia concentration is

shown as a function of the AC dose, ranging from 0 to 3 g/200 mL while using a fixed dose of 1 g/L nZVI.

Additionally, another set of experiments where the samples were first treated with 1 g/L nZVI, followed by the addition of the nZVI-AC composite as illustrated in **Fig. 7**. The residual nitrate and ammonia concentrations after this two-step treatment process are also provided.



**Fig. 4:** XRD patterns for the nonactivated carbon (A) and activated carbon (B), respectively.

The introduction of activated carbon (AC) alongside nZVI nanoparticles aimed to investigate the potential synergetic effect of combining these two materials for enhanced nitrate removal and minimizing the formation of undesirable by-products, such as ammonia.

The results demonstrate that the nZVI-AC composite effectively reduced nitrate concentrations, with the residual levels varying based on the AC dose. The presence of AC likely contributed to the adsorption of nitrate ions, complementing the reduction capability of the ZVI nanoparticles.

The formation of ammonia as a side product during the nitrate removal process is a common phenomenon when using ZVI nanoparticles. The ammonia concentration was found to decrease with increasing AC doses, suggesting that AC plays a role in adsorbing or minimizing the formation of ammonia. This is particularly beneficial as ammonia can be a potential contaminant in treated water.

The two-step treatment process, where the samples were first treated with nZVI and then with the nZVI-AC composite,

showed further reductions in both nitrate and ammonia concentrations. This approach likely allowed for the initial reduction of nitrate by nZVI, followed by the adsorption of any remaining nitrate and ammonia by the AC in the composite. The adsorption capacity of AC for nitrate ions was also investigated separately by conducting batch experiments with varying initial nitrate concentrations and a fixed AC dose. The results indicated that AC alone could effectively adsorb nitrate ions, further contributing to the overall nitrate removal performance observed in the nZVI-AC composite system.

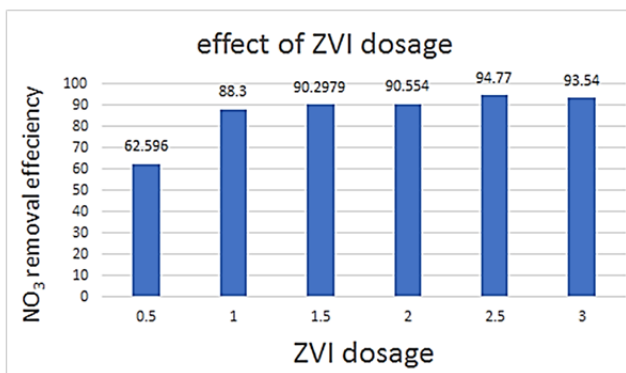


Fig. 5: Nitrate removal efficiency of ZVI nanoparticles.

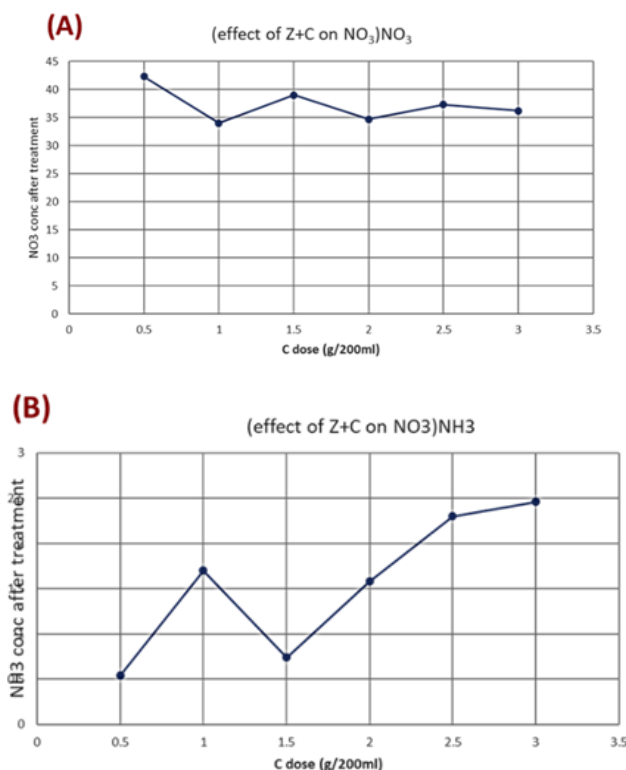


Fig. 6: Nitrate removal efficiency of nZVI and activated carbon composites (A), and ammonia removal (B).

The combination of nZVI and activated carbon exhibited a synergetic effect in enhancing the removal of nitrate ions from aqueous solutions. The nZVI acted as a reducing agent, facilitating the conversion of nitrate ions, while the porous structure and high surface area of AC contributed to the adsorption of nitrate ions and potential by-products like

ammonia. The synergetic effect can be attributed to the complementary mechanisms of reduction (by nZVI) and adsorption (by AC), which allowed for efficient nitrate removal and minimization of undesirable by-products. The ability to control the formation of ammonia is particularly important for ensuring the quality of treated water and minimizing the potential environmental impact.

Overall, the results demonstrate the promising potential of the nZVI-AC composite system for effective nitrate remediation in water treatment applications. The combination of these two materials leverages their respective strengths and offers a more comprehensive approach to nitrate removal and by-product management.

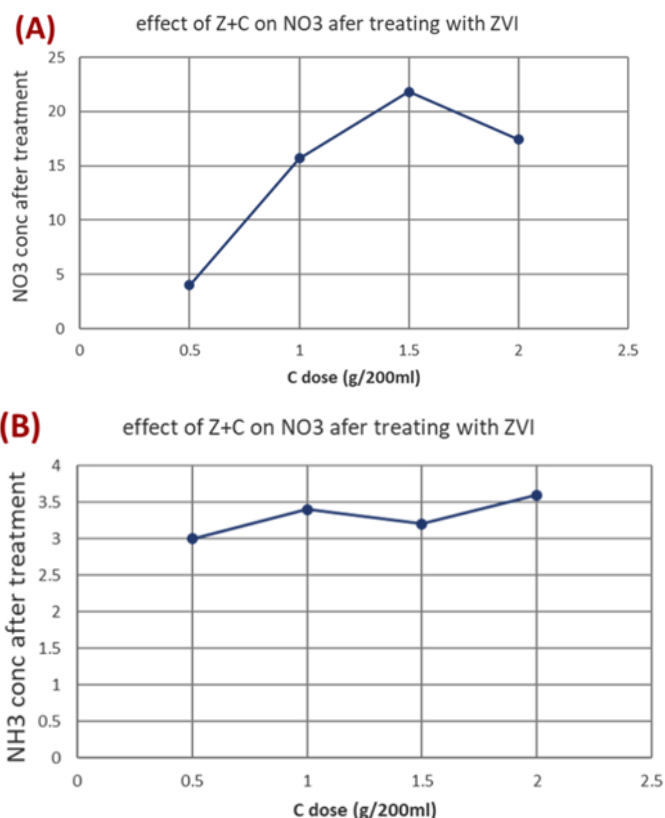


Fig. 7: Nitrate removal efficiency of nZVI and activated carbon composites (A), and ammonia removal (B), as two separate steps.

#### 4. Conclusion

This study successfully demonstrated the synthesis and application of nZVI and AC for the effective removal of nitrate ions from polluted potable water sources. The synergetic combination of these two materials exhibited superior performance in nitrate remediation compared to their individual use. The synthesized nZVI, characterized by SEM, TEM, EDX, and XRD analyses, exhibited a spherical morphology with an average particle size of 40 nm and a highly crystalline structure. These nanoparticles displayed excellent reducing capabilities, effectively converting nitrate ions through an electron transfer mechanism. Batch experiments revealed that the nZVI alone achieved high nitrate

removal efficiency, with increasing dosages leading to improved performance. The introduction of AC further enhanced the nitrate removal process by exploiting its adsorption properties and complementing the reduction capability of nZVI. The synergetic effect of the nZVI-AC composite was evident in the significant reduction of nitrate concentrations and the minimization of ammonia formation, a common by-product of nitrate reduction processes. The combination of reduction by nZVI and adsorption by AC allowed for efficient nitrate removal while mitigating the generation of undesirable by-products. Moreover, the two-step treatment approach, where samples were first treated with nZVI and then with the nZVI-AC composite, demonstrated further improvements in nitrate and ammonia removal, highlighting the complementary roles of these materials in the treatment process. The adsorption capacity of AC for nitrate ions was also evaluated, confirming its potential as a standalone adsorbent for nitrate removal, albeit with lower efficiency compared to the nZVI-AC composite system. The synergetic effect of nZVI and AC offers a promising approach for efficient nitrate remediation while minimizing the formation of undesirable by-products, addressing a critical environmental issue, and contributing to the development of sustainable water treatment technologies. Furthermore, this study's findings have significant practical implications, particularly in addressing water pollution and providing cleaner potable water. The novel approach demonstrated here can be scaled and adapted for broader applications in industrial and municipal water treatment facilities. Future perspectives include the exploration of more advanced nanocomposite materials and optimizing the process for even greater efficiency. The potential for integrating this technology with existing water treatment systems underscores its relevance and utility in real-world applications. As part of our ongoing research, we plan to explore the potential of these materials in other environmental and industrial applications, such as battery technology, where their unique properties can be further exploited. This aligns with Egypt's 2030 development strategy to promote sustainable green energy solutions and contribute to global efforts in combating climate change.

### CRedit authorship contribution statement:

Conceptualization, Mahmoud Elrouby and Alaa ElFenjary; methodology, Alaa ElFengary; software, Hany Abdelateef; validation, Hossnia Mohran, and Mahoud Elrouby.; formal analysis, Mahmoud Elrouby; investigation, Hany; writing—original draft preparation, Mahmoud Elrouby; writing—review and editing, Hany.; visualization, Hany; supervision, Hossnia. All authors have read and agreed to the published version of the manuscript.

### Data availability statement

The data used to support the findings of this study are available from the corresponding author upon request.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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