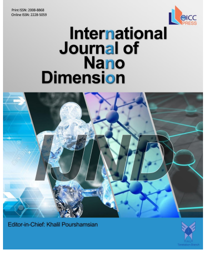

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Original Research

RSM-CCD Optimized EDTA-Functionalized Magnetic Activated Carbon Derived from Sunflower Stem for Adsorptive Removal of Ni^{2+} and Malachite Green from Containment Water

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Abstract:

The structure of malachite green (MG) molecules makes them highly resistant to environmental degradation, posing significant challenges for removal. Similarly, heavy metals such as Ni^{2+} present substantial risks due to their toxicity and persistence. The presence of MG and Ni^{2+} in water and wastewater is frequently reported at levels exceeding standard limits, highlighting the urgent need for effective removal and degradation strategies. Herein, EDTA-functionalized magnetic activated carbon (EDTA-FMAC) derived from sunflower stems was developed and successfully applied for the removal of MG and Ni^{2+} from contaminated water. The adsorbent (MAC) was characterized using FTIR, XRD, SEM, Raman, VSM, and BET techniques. Key factors, including sorbent weight, contact time, initial pollutant concentration, and pH, were optimized using Response Surface Methodology (RSM). The study achieved maximum adsorption efficiencies of 97.68% for MG and 94.63% for Ni^{2+} at initial concentrations of 15 and 20 mg/L, adsorbent weights of 9 and 20 g, contact times of 25 and 20 minutes, and pH levels of 6 and 4, respectively. The experimental data aligned closely with the Freundlich isotherm model and the pseudo-second-order kinetic model. Thermodynamic analysis indicated that MG and Ni^{2+} adsorption onto magnetic activated carbon is endothermic and spontaneous. This study demonstrates that EDTA-functionalized magnetic activated carbon derived from sunflower stems is an efficient, effective, and reusable adsorbent for the removal of MG and Ni^{2+} from tap water.

Keywords: Adsorption; Functionalized activated carbon; Heavy metals; Magnetic activated carbon; Malachite green

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1. Introduction

Nickel, a toxic heavy metal, is widely used in various manufacturing sectors, including metal plating, galvanizing, smelting, mining, pigment production, and ceramics. Consequently, it is frequently found in industrial effluents [1, 2, 3, 4, 5, 6]. This non-biodegradable metal has a high potential for accumulation in microorganisms, potentially leading to dermatitis and allergic reactions [7]. At higher concentrations, nickel is a potent carcinogen, associated with cancers of the lungs, nose, and bones [8, 9, 10]. With a pKa of approximately 9.86 for its hydrated form, Ni^{2+} remains in its cationic state under acidic to near-neutral pH conditions, which supports its mobility and availability for adsorption. Therefore, the removal of nickel from industrial effluents is crucial [11].

In parallel, environmental concerns also extend to malachite green (MG), a cationic dye widely used in various industrial sectors as a coloring agent. MG is released into water during its synthesis and application processes [12]. With a *pKa* of approximately 6.9, it primarily exists in its protonated cationic form under typical environmental pH conditions. Known for its toxicity and carcinogenicity, MG can cause skin irritation, allergies, and cancer in humans, underscoring the urgent need for its removal from water sources [13, 14].

To date, numerous techniques have been employed to purify polluted water and remove metal ions and dyes, including electrochemical methods, chemical precipitation, membrane filtration, ion exchange, and adsorption [12, 15, 16]. Among the diverse purification techniques, adsorption has emerged as one of the most reliable and technically feasible approaches due to its simplicity, ease of preparation and application, reproducibility, and cost-effectiveness [17, 18]. Common adsorbents include activated carbon, zeolites, chitosan, graphene, polymer materials, and clay. However, despite these advances, a critical gap remains: few studies have successfully developed a sustainable adsorbent that combines dual-function pollutant removal, high reusability, and magnetic separability using an eco-friendly synthesis route [19, 20]. As a result, there is a pressing need to develop versatile, high-capacity adsorbents capable of treating diverse pollutants in contaminated water. Addressing this challenge is critical for many countries facing serious water pollution issues [21, 22].

The use of activated carbon (AC) for heavy metal adsorption offers numerous advantages, including high adsorption capacity, effective metal recovery, excellent selectivity, sludge-free operation, and cost-effectiveness [23, 24]. However, the widespread application of commercial activated carbon is limited due to its production from expensive and non-renewable sources such as oil residues, wood, coal, and peat [25]. Recently, there have been advances in using abundant agricultural waste as a renewable, cost-effective alternative, rich surface functional groups and abundant inorganic mineral for AC production [26, 27]. Sunflower, extensively cultivated across many countries, generates significant waste in the form of stems [28]. These stems, primarily composed of carbonaceous compounds ($40-50\%$ cellulose, $20-30\%$ hemicellulose, and $15-20\%$ lignin), have been identified as excellent precursors for producing activated carbon [29]. Several studies have demonstrated the successful production of inexpensive and efficient powdered and granular activated carbon from sunflower stems through chemical activation methods [30, 31]. The resulting activated carbon exhibits a high surface area and porous structure, making it suitable for various applications, including wastewater treatment, water purification, and air filtration technologies [32]. However, one of the key challenges is the hydrophobic nature of activated carbon, which hinders its performance in water purification. To address this issue, chemical surface modification is often necessary. The addition of hydroxyl or carbonyl functional groups can enhance the hydrophilicity of activated carbon. Furthermore, inducing magnetic properties is an effective strategy to improve adsorption capacity, efficiency, and facilitate the easy separation of the adsorbent from water sources [33, 34, 35, 36].

Ethylenediamine tetraacetic acid (EDTA), a polycarboxylic amino acid, has a strong ability to form stable chelates with metal ions and is considered a cost-effective material, making it widely used for modifying various substances. One of the key advantages of applying EDTA to different materials is its ability to enhance the adsorption of metal ions. However, research indicates that the adsorption capacity of EDTA-modified materials is significantly influenced by the type of substrate used [37, 38]. Among the various substrates, activated carbon (AC) stands out due to its unique properties, making it a highly effective and widely used adsorbent [39].

Fe_3O_4 nanoparticles are widely recognized as effective magnetic materials due to their simple preparation process and excellent biocompatibility. The magnetization of activated carbon (AC) sheets with Fe_3O_4 nanoparticles has driven the development of magnetic adsorbents for pollutant removal [40, 41].

To address this gap, the present work aims to develop a cost-effective, reusable, and magnetically responsive bio-based adsorbent capable of efficiently removing both Ni^{2+} and MG from wastewater. The AC was subsequently magnetized and functionalized with EDTA, serving as a chelating agent. The morphology, structure, and magnetic properties of the synthesized EDTA-functionalized magnetic activated carbon (EDTA-FMAC) were characterized through various analyses. The adsorption capacity of EDTA-FMAC for Ni^{2+} and MG was investigated. Additionally, the reproducibility and reusability of EDTA-FMAC were evaluated, demonstrating that the material is an efficient, stable, and reusable adsorbent for the removal of Ni^{2+} and MG from contaminated water. While some adsorbents reported in the literature exhibit high adsorption capacities such as banana peel-derived biosorbents (388.9 mg/g) [42], CNT/alumina composites (229.9 mg/g) [43], and Na-P1 zeolites (416.6 mg/g) [44] these materials often involve complex synthesis methods or limited reusability. Similarly, bottom ash achieved a malachite green adsorption capacity of 173.9 mg/g [45], though its recovery from aqueous systems remains challenging. In comparison, the EDTA-FMAC developed in this work displayed excellent adsorption capacities of 157.72 mg/g for Ni^{2+} and 162.80 mg/g for MG under optimized conditions. Although numerically slightly lower, these values were achieved using an abundant agricultural waste source, through a straightforward and eco-friendly functionalization route. The material demonstrated not only high dual-function adsorption performance but also magnetic separability and superior reusability, making it a compelling, scalable candidate for real-world wastewater treatment applications. This study addresses the critical need for sustainable and multifunctional adsorbents by developing a bio-derived material capable of removing both heavy metals and dyes from wastewater. The main objective is to synthesize and characterize an EDTA-functionalized, magnetically separable activated carbon from sunflower stem waste, and to evaluate its adsorption performance, reusability, and stability. This work not only contributes to the advancement of green adsorbent materials but also opens avenues for cost-effective and scalable solutions in environmental remediation, with potential applications in industrial-scale water treatment systems.

2. Experimental

2.1 Materials

All chemicals used in this study were of analytical grade and used without further purification. Ammonia solution 25% (M/s Merck, Germany), ethanol 96% (M/s Kimia Alcohol Zanjan, Iran), iron(II) sulfate heptahydrate (M/s Merck, Germany), ethylenediamine tetraacetic acid (EDTA) (M/s Merck, Germany), sodium hydroxide (M/s Kian Kave Azma, Iran), malachite green dye (M/s Merck, Germany), and nickel(II) nitrate 99.5% (M/s Merck, Germany) were purchased and used as received. Sunflower stems were collected from farms in Sufiyan (Tabriz, Iran). Potassium hydroxide, additional sodium hydroxide, acetic acid 96% (all from M/s Merck, Germany), and ferric chloride hexahydrate (M/s Sigma-Aldrich) were also employed in this research.

2.2 Instruments

The FT-IR spectrophotometer (Tensor 27, Bruker) was used to record the spectra by KBr pellets. X-ray diffraction (XRD) measurements were performed at room temperature by a Siemens diffractometer using Cu-Kα radiation at 35 kV in the scan range of 2θ from 5 to 80° (Tongda, TD-3700). UV–Vis absorption spectra were performed on a Shimadzu 1700 Model UV–Vis spectrophotometer. Scanning electron microscopy (SEM) (MIRA3 FEG-SEM Tescan, Czech) was used for analyzing morphology of samples and the magnetization behavior was measured by the magnetometer (VSM, model MDKF, Iran). Raman spectrum was determined by trchnooran company instrument (model Ram-532-004) in 532 nanometer wavelength.

2.3 Synthesis of sunflower stems based activated carbon

The creation of activated carbons from sunflower stem materials involves a two-step process; it starts with carbonization at low temperatures (700 – 800 ºK) in an oxygen-free environment to remove volatile substances, followed by activation at higher temperatures (1100 – 1300 ºK) to enhance the porosity and surface area of the resulting solid. In this study Sunflower stems were cut into small pieces and dried on oven at 100 °C, then it was crushed as a uniform powder, the obtained powder was ground and screened to collect average size of AC particles. Then the activating agents KOH were added to the powder. For this purpose, 1.5 grams of powder was soaked in 20 mL of 30% KOH. The ratio of active carbon to the base was 1:3; it was stirred for 10 hours at room temperature. The activation process was carried out in a vacuum oven at 700 °C for 1 hour at 10 °C/min. Then, the activated carbon was dried after washing several time with ethanol: water, to reach the neutral pH range. Finally, the sunflower stem-based activated carbon (SSAC) was dried in an oven at 80 °C to obtain the product [46, 47].

2.4 Magnetization of sunflower stems based activated carbon

The co-participation method was employed to synthesize Magnetite Activated Carbon (MAC) nanocomposite. To initiate the magnetization process, 2.5 g of activated carbon obtained previous method was combined with 500 mL of distilled water and ultra-sonicated while 1 g of $FeCl_3 \cdot 6H_2O$ and 0.4 g of $FeSO_4 \cdot 7H_2O$ were added at a temperature of 80 °C. Ammonia solution was then added dropwise to adjust the pH to 9, The mixture was stirred in an inert environment at 60 °C for 2 hours to allow for deoxygenation. Following this, the solid particles were magnetically separated, rinsed three times with a 1:1 solution of water: ethanol, and ultimately dried at 60 °C for 24 hours [48, 49].

$$FeSO_4 + 2FeCl_3 + 8NH_4OH^- \rightarrow Fe_3O_4 + 6NH_4Cl + (NH_2)_2\,SO_4 + 4H_2O$$

2.5 EDTA- functionalized sunflower stems based activated carbon

To prepare the EDTA-FMAC adsorption, 4.5 g of EDTA and 3 g of NaOH were mixed with the magnetic activated carbon obtained from the previous step in 150 mL of distilled water. The mixture was stirred uniformly at ambient temperature for 24 hours. Afterward, the resulting suspension was washed with a 1:1 ratio of ethanol and distilled water. Finally, the mixture was dried at 60 °C [50, 51].

2.6 Adsorption studies

Adsorption and isotherms were investigated by conducting batch experiments and analyzing via AAS instrument. The adsorption capacity at equilibrium (q_e, mg/g), and the percentage removal (Removal, %) can be calculated by the following equations:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

$$\text{Removal\%} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

where q_e is the adsorption capacity (mg/g), C_0 (mg/L) and C_e (mg/L) are the initial and equilibrated concentrations, respectively, V (L) is the volume of solution added, and m (g) is the mass of the adsorbent [52].

2.7 Optimization of the adsorption process of Ni^{2+} and MG via RSM

In this study, RSM, the Central Composite design method (CCD), is used to optimize the absorption process for the removal of Ni^{2+} ions and MG. Four effective Factors in the process of Ni^{2+} ions and MG removal, including the initial concentration of Ni^{2+} and MG (mg/L), the initial

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pH of the Ni^{2+} and MG solution, the absorption time (min) of Ni^{2+} and MG, and the adsorbent weight (mg) were investigated (Table 1). 31 experiments were proposed by Design Expert 11, which included 6 repetitions at the central point. The following second-order multi-order equation was used to connect between dependent and non-dependent variables:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{1 \le i \le j}^{k} \beta_{ij} X_i X_j + \varepsilon \quad (3)$$

where (Y) represents the adsorption efficiency, X_i and X_j denote the experimental levels of variables, (β_i) stands for the linear correlation coefficient, (β_{ii}) indicates the second-order correlation, (β_{ij}) signifies the cross-correlation coefficient, and (ε) represents the random error. The variables i, j, and k correspond to the number of variables under analysis.

2.8 Adsorption isotherms and reaction kinetics studies

Adsorption behavior is commonly interpreted using theoretical models that describe both equilibrium and kinetic aspects of the process. Isotherm models such as Langmuir and Freundlich are used to characterize the interaction between adsorbate molecules and the surface of the adsorbent. These models help determine whether adsorption occurs as a monolayer on a homogeneous surface or as multilayer coverage on heterogeneous sites. Kinetic models, including pseudo-first-order and pseudo-second-order equations, are employed to evaluate the rate and mechanism of adsorption, indicating whether the process is governed by physical diffusion or chemical interaction. The linear forms of these models, along with their corresponding plotting formats, are summarized in Table 2. These equations provide the basis for analyzing experimental data and optimizing adsorption systems for practical applications.

Where k_1 and k_2 represent the rate constants for the pseudo-first-order and pseudo-second-order kinetic models, respectively (h^{-1}). The terms q_e and q_t denote the amount of adsorbate retained by the adsorbent at equilibrium and at a given time t, respectively, both expressed in mg/g. The variable C_e refers to the equilibrium concentration of the contaminant in solution (mg/L), while q_m indicates the theoretical maximum adsorption capacity under equilibrium conditions (mg/g). The Langmuir constant K_L is associated with the affinity of binding sites and the energy of adsorption. In the Freundlich model, K_F and $1/n_1/n$ are empirical constants that reflect the adsorption capacity (mg/g) and the intensity or favorability of the adsorption process, respectively.

3. Results and discussion

3.1 Characterization

Figure 1 exhibits FT-IR spectra of AC, MAC and EDTA F-MAC derivatives presented from bottom to top. A sharp peak observed at 3435.98, 3431.46 and 3426.65 cm^{-1} is related to stretching vibration of OH functional group (3200 – 3500 cm^{-1}) respectively which assigns OH stretching vibration of phenols and water molecules. Distinct peaks at 2920.94, 2924.81 and 2922.28 cm^{-1} represent aliphatic stretching vibration related to CH_2 group (2800 – 3000 cm^{-1}). The sharp peak at 1639.21, 1643.81, and 1642.4 cm^{-1} is attributed to the carbonyl (C=O) functional group (1600 – 1650 cm^{-1}). The Fe–O stretching vibration peak for MAC appears at 581.73 cm^{-1}, which shifts to 557.36 cm^{-1} in EDTA-FMAC due to the carbonization effect. A distinct peak observed at

Table 1. Range of parameters and levels of test variables for Pb^{2+} removal.

Variable	Levels				
	−2	−1	0	1	2
Ni^{2+} Concentration (X_1)	5	10	15	20	25
Initial pH (X_2)	3	5	7	9	11
Adsorbent weight (X_3)	3	5	7	9	11
Time (X_4)	5	15	25	35	45

Table 2. Scientific principles and modeling approaches in adsorption studies.

Kinetic models	Linear form	Plot
Pseudo-first order	$\frac{dq_t}{dt} = K_1(q_e - q_t)$	$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t$
Pseudo-second order	$\frac{dq_t}{dt} = K_2(q_e - q_t)^2$	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$
Isotherm models	Linear form	Plot
Langmuir	$q_e = \frac{c_e k_l q_m}{1 + c_e k_l}$	$\frac{c_e}{q_e} = \frac{1}{k_1 q_m} + \frac{c_e}{q_m}$
Freundlich	$q_e = K_f C_e^{1/2}$	$\log q_e = \frac{1}{n} \log C_e + \log K_f$

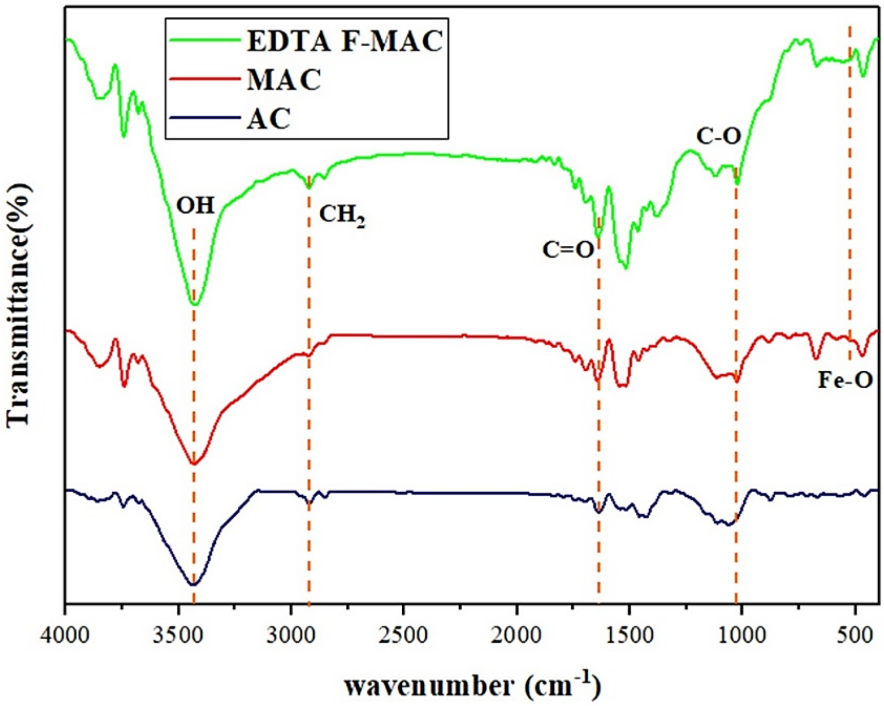

Figure 1. The FTIR spectrum of EDTA F-MAC, MAC and AC.

1061.48, 1025.12, and 1023.35 cm^{-1} corresponds to the C–O stretching vibration (1018 – 1149 cm^{-1}). These characteristic peaks confirm the successful synthesis and accuracy of the adsorbent [53, 54, 55].

Figure 2 presents the Raman spectra of AC sample, MAC and EDTA F-MAC, respectively. All of samples display common broad peaks approximately at 1320 – 1340 and 1580 – 1590 cm^{-1}, corresponding to D bond and G bond. G bond indicates sp^2 C-C in-plane vibrations and graphite lattice. D bond exhibits sp^3 structural defects which show disorders of active sites. I_D/I_G is a ratio for representing structures disorder in graphitic materials [56, 57]. This ratio for AC, MAC and EDTA F-MAC is 1.01, 1.03 and 1.04 which claims enhancement the defects of modified AC adsorbent leading to increase the active sites for removal of pollutants. Three peaks at 214, 284 and 398 cm^{-1} for Fe_3O_4 were identified in MAC spectra. 214 and 284 were considered to E_g and

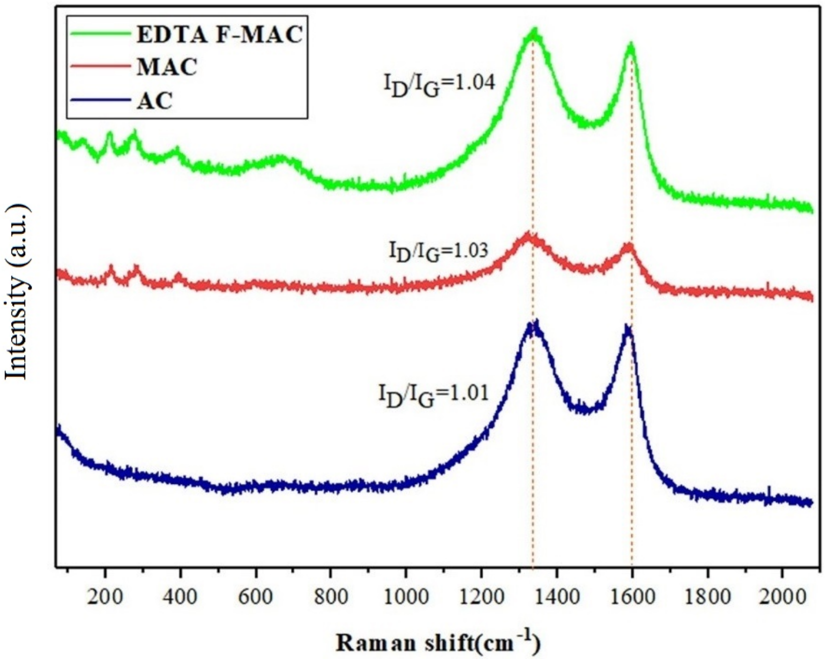

Figure 2. The raman shift of EDTA F-MAC.

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T_{2g} asymmetric modes and 398 was corresponded to A_{1g} mode [58, 59].

XRD analysis is one of the best techniques to identify the crystallinity or amorphous structure of ACs derived materials. The Fig. 3 shows XRD graph of AC which exhibits 2 sharp peaks at around $2\theta = 26.8°$ and $2\theta = 44.7°$ corresponding to (002) and (101) planes for thick layers of carbon-containing structure. These peaks demonstrate the effect of employed temperature for activation of AC which lead to formation of the partial graphitic structure at amorphous AC. According to JCPDS card (JCPDS-19-0629) analysis of MAC in comparison with AC exhibits diffraction peaks related to (311), (400), (440) cubic crystal planes of Fe_3O_4 penetrating to AC structure. The diffraction peak at around $2\theta = 17.02°$ probably is related to EDTA structure [60, 61].

The FESEM analysis is an appropriate method to illustrate the morphological details of MAC and EDTA-functionalized MAC, as shown in Fig. 4. Figures 4 (a,b) depict a relatively smooth structure with some pores and surface wrinkles on the MAC. After the functionalization process with EDTA, it is evident that EDTA is well-dispersed within the MAC, leading to significant changes in the heterogeneous structure and porosity. This modification results in spherical agglomeration of particles, enhancing the porous structure and improving the adsorbent properties [62, 63, 64].

Figure 5 shows the N_2 adsorption-desorption isotherm for the EDTA F-MAC adsorbent. The graph is classified as a type I isotherm, indicating the presence of two-dimensional slit pores. The incorporation of Fe_3O_4 particles into the AC pores, along with EDTA within the MAC structure, reduces N_2 adsorption capacity. Noticeable values like as, V_m and total pore volume are 146.85 m^2/g, 33.739 cm^3/g and 0.1333 cm^3/g, respectively. Additionally, Fig. 6 presents the pore volume distribution of the synthesized adsorbent. The peak pore radius (r_p) for EDTA F-MAC is 1.21 nm, indicating a microporous structure (< 2 nm). This microporosity plays a significant role in the sharp increase in N_2 adsorption [65, 66].

The magnetic property versus applied magnetic field of EDTA F-MAC is exhibited on Fig. 7. The synthesized adsorbent, with a saturation magnetization (µs) of 8.15 emu/g, shows superparamagnetic characteristics. The sigmoidal curve and the ease of particle separation with a magnet highlight the adsorbent's suitability for use in aqueous solutions [67, 68, 69].

3.2 Analysis of variance (ANOVA)

Various RSM computations for the current optimization study were performed employing Design-Expert 11 software. A statistical second-order model, which includes interaction and polynomial terms, was developed for all response variables. The general form of the model is represented as in the following:

$$Y = 73.29 - 5.8X_1 - 2.35X_2 + 3.96X_3X_4.65X_1X_2 - 2.27X_1X_3 + 4.81X_2X_3$$

$$Y = 76.28 - 0.91X_1 - 1.96X_2 - 3.27X_3 + 9.61X_4 - 1.07X_1X_3 + 1.90X_2X_3 - 1.69X_2X_4 - 0.69X_3X_4 + 0.69X_4^2$$

These equations represent the statistical model, where Y is the response variable and X_1, X_2, X_3, and X_4 are the independent variables. The model includes both interaction and polynomial terms to analyze the complex interactions between the independent variables and their effects on the response variable.

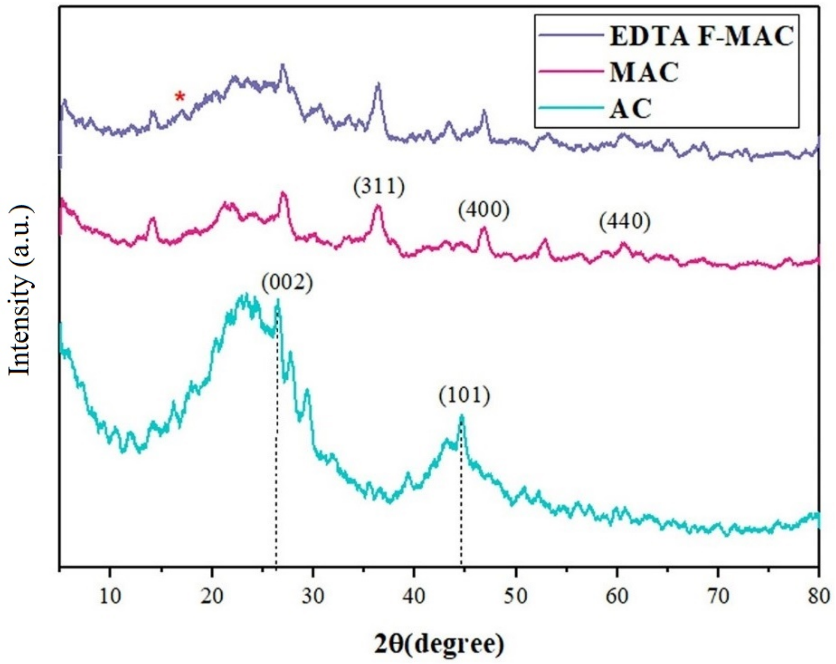

Figure 3. The XRD pattern of EDTA F-MAC, MAC and AC.

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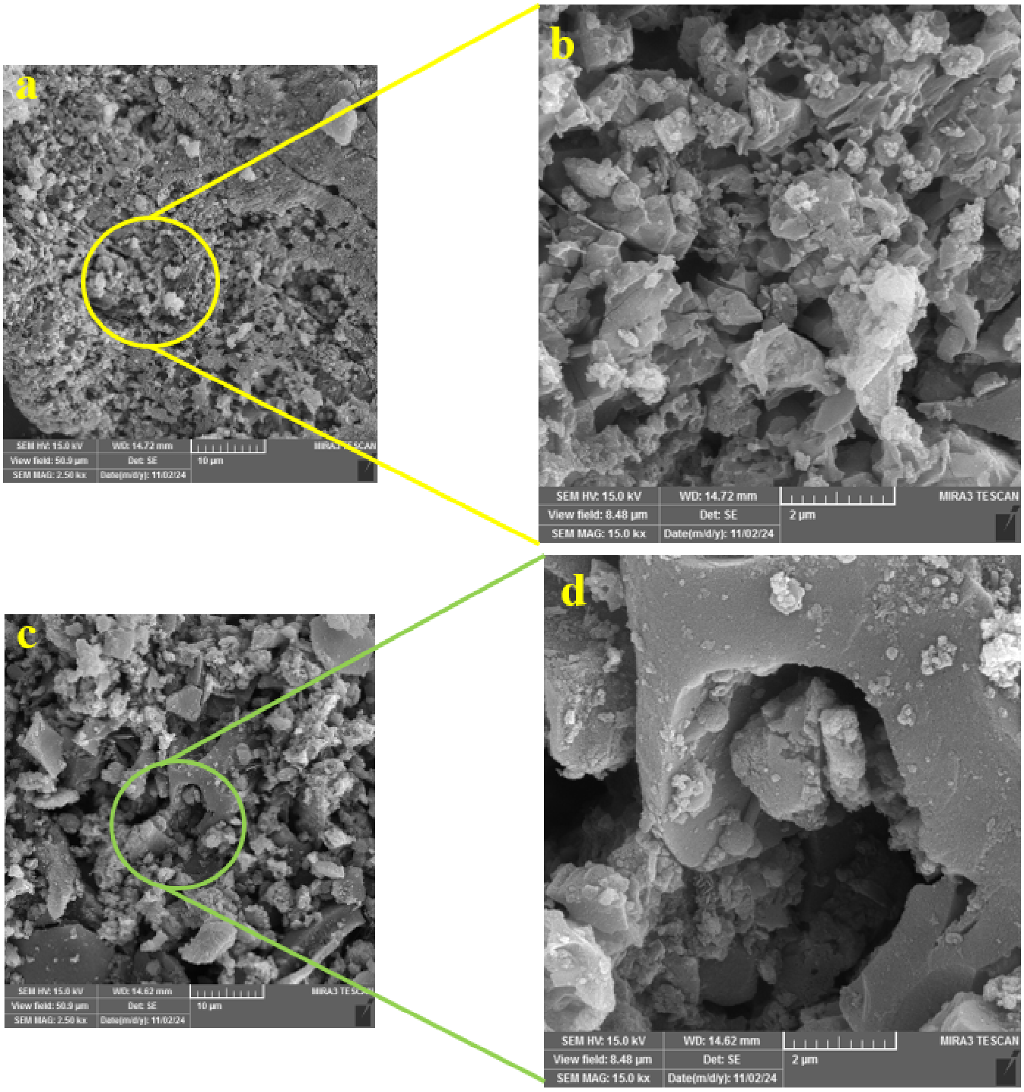

Figure 4. (a, b, c, d): The SEM images of MAC and EDTA F-MAC.

Coefficients of the variables (e.g., $-5.8X_1$ and $3.96X_3X_4$) indicate the influence of each variable on the final response. Interaction terms (such as X_1X_2 and X_2X_3) reflect the combined effects of two or more independent variables on the response variable. Using such advanced statistical models allows for a precise analysis of the individual and interactive effects of independent variables on the response, helping to optimize the study's results. Table 3 presents both the experimental data and the results derived from the model equations. The findings demonstrate that this model accurately represents the experimental outcomes and is effective in predicting the output variable under various conditions. A comparison between the experimental results for the adsorption efficiency of Ni2+ and MG and the RSM model outcomes shows a significant overlap, as visually represented in Figs. 8 (a,b). Additionally, the ANOVA results are provided in Table 4. Based on these results, the proposed model is identified as quadratic.

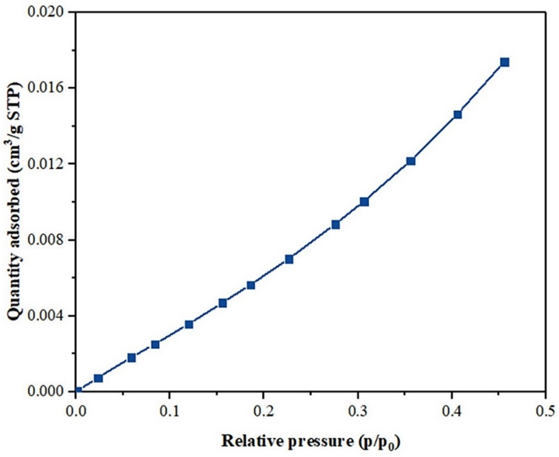

Figure 5. N_2 adsorption-desorption isotherm for EDTA F-MAC.

3.2.1 Effect of pH and pollutant concentration on adsorption efficiency and point of zero charge (pHpzc)

To determine the point of zero charge (pHpzc) of the EDTA-FMAC adsorbent, a series of 50 mL solutions of 0.1 N $NaNO_3$ were prepared. The initial pH of each

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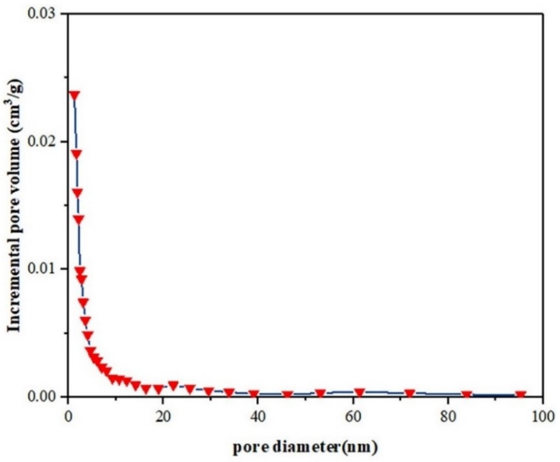

Figure 6. Pore volume distribution of EDTA F-MAC.

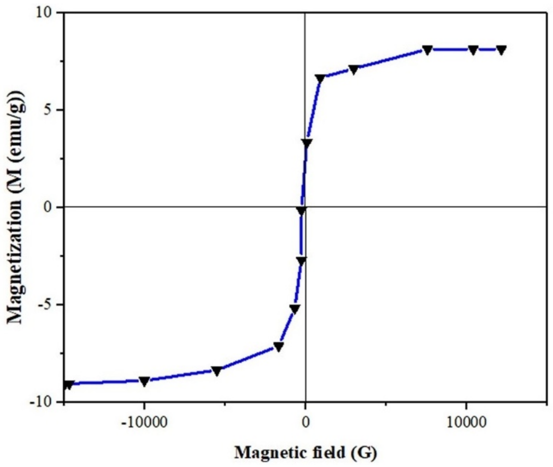

Figure 7. The magnetic property versus applied magnetic field of EDTA F-MAC.

solution was adjusted to 2, 4, 6, 8, 10, and 12 using 0.1 N HCl or NaOH. Then, 40 mg of the adsorbent was added to each solution, and the suspensions were stirred at room temperature for 48 hours to ensure equilibrium. After equilibration, the final pH was measured, and the difference between final and initial pH (ΔpH) was calculated. The ΔpH values were plotted against the initial pH to determine the pHpzc. The point at which ΔpH = 0 was identified as the pHpzc, which was approximately

Table 3. Experimental and RSM predicted results for Ni^{2+} adsorption.

	Factor 1	Factor 2	Factor 3	Factor 4	Response 1	Response 2
Run	A:pH	B:Pollutant Concentration	C:Adsorbent weight	D:time	Ni	MG
1	4	20	6	10	82.23	66.67
2	6	5	9	15	78.25	80.32
3	6	15	9	25	81.19	97.68
4	4	20	6	20	80.22	85.47
5	8	20	6	20	46.31	86.45
6	8	10	6	20	70.1	95.64
7	8	10	12	20	80.65	83.29
8	8	10	6	10	80.62	72.26
9	4	20	12	10	70.26	69.12
10	4	10	12	20	81.85	87.54
11	8	20	6	10	54.62	68.12
12	4	20	12	20	94.63	83.07
13	8	20	12	10	60.83	65.89
14	6	15	9	15	73.21	75.98
15	6	15	3	15	72.23	84.52
16	4	10	6	10	84.49	72.69
17	6	15	15	15	71.62	69.8
18	8	20	12	20	78.59	79.28
19	6	15	9	15	74.19	75.98
20	2	15	9	15	82.21	79.09
21	6	15	9	15	74.19	75.98
22	6	15	9	15	72.41	77
23	4	10	6	20	86.92	95.78
24	8	10	9	15	70.14	71.18
26	4	10	12	10	57.04	64.85
27	6	15	9	15	73.07	76.77
28	6	15	9	5	64.96	60.36
29	10	15	9	15	62.35	74.84
30	6	15	9	15	73.04	75.98

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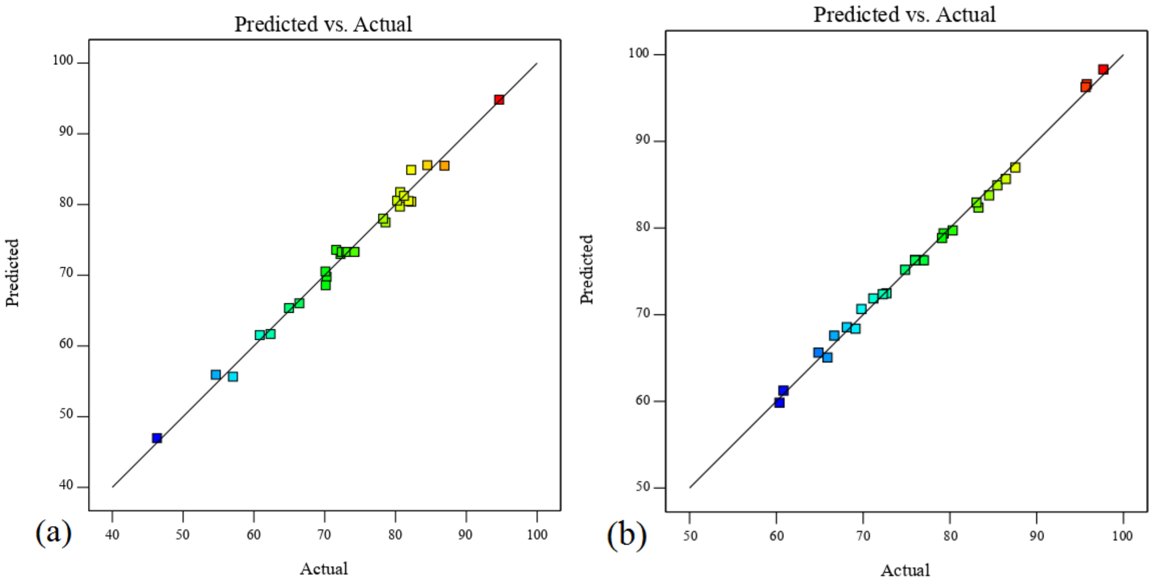


Figure 8. Comparison of predicted and actual value for removal of (a) Ni^{2+} and (b) MG based on RSM results.

Table 4. ANOVA analysis of Ni^{2+} and MG removal.

Source of variations	Regression		Adjusted R2		Predicted R2		F-value		Lack of Fit		Model	
	R1	R2	R1	R2	R1	R2	R1	R2	R1	R2	R1	R2
	0.98	0.99	0.98	0.96	0.99	0.98	166.60	282.37	0.0517	0.0651	2FI	Quadratic

4.0 for Ni^{2+} and 6.5 for MG (Figs. 9 (a,b)). The pH of the solution plays a crucial role in controlling the surface charge of the adsorbent and, consequently, the adsorption behavior of both Ni^{2+} and MG. At pH values below the pHpzc, the adsorbent surface becomes positively charged, which typically repels cationic species. However, Ni^{2+} adsorption is enhanced under acidic conditions (pH $\approx$ 4) due to strong chelation with the EDTA functional groups, which dominate over electrostatic repulsion. In contrast, MG adsorption is most efficient near pH 6.5, where the surface charge is neutral to slightly negative, allowing favorable interactions with the dye's aromatic rings and polar functional groups such as hydroxyl and carbonyl. A comparative analysis across the same pH range reveals distinct adsorption mechanisms for the two pollutants. Ni^{2+}, as a small divalent metal ion, is highly sensitive to electrostatic interactions and forms stable complexes with EDTA, leading to optimal adsorption at lower pH. MG, being a bulky organic dye, interacts primarily through $\pi - \pi$ stacking and hydrogen bonding, which are favored near neutral pH. These differences in molecular structure and interaction pathways explain the observed variation in adsorption efficiency between N^{2+} and MG [70].

The 3D graph for Ni^{2+} and MG removal based on RSM provided in figures 10 (a-l). As shown in Fig. 10 (a) (for Ni^{2+} removal) the efficiency initially increases with rising pH. However, beyond pH 6, the removal efficiency begins to decrease. This decline could be attributed to the formation of soluble complexes or other chemical interactions that negatively impact the Ni^{2+} removal process. At lower pH values, increasing the pollutant concentration appears to enhance removal efficiency. This could be due to the increased availability of adsorption sites or favorable chemical interactions at lower pH, which promote better adsorption of Ni^{2+}. For MG, as shown in Fig. 10 (g), lower pH and higher pollutant concentration lead to improved removal efficiency. Acidic conditions can enhance adsorption capacity, and a lower pollutant concentration reduces competition for adsorption sites, thus improving overall efficiency.

3.2.2 pH and adsorbent weight effect on adsorption efficiency

As shown in Fig. 10 (b), the interaction between pH and adsorbent weight significantly affects the removal efficiency of Ni^{2+}. Increasing the adsorbent weight at lower pH values leads to enhanced adsorption, primarily due to the greater availability of active sites and increased surface area. A higher dosage allows more pollutant molecules to interact with functional groups such as carboxyl and EDTA moieties, improving overall efficiency. At higher pH levels (above 6), the formation of nickel hydroxide species such as $Ni(OH)_2$ becomes dominant. These complexes are poorly soluble and tend to precipitate, reducing the concentration of free Ni^{2+} ions in solution. Since the adsorbent primarily interacts with ionic forms of nickel, the presence of hydroxide complexes inhibits adsorption. This phenomenon explains the observed decline in efficiency at elevated pH values. The 3D response surface plot (Fig. 10 (b)) illustrates this trend clearly: maximum adsorption occurs at low pH (≈ 4) and high adsorbent weight (≈ 12 mg). The curvature of the surface confirms a nonlinear relationship, where adsorption efficiency increases sharply with adsorbent dose at acidic pH but plateaus or declines at higher pH due to chemical speciation effects. A similar pattern is observed for MG adsorption (Fig. 10 (h)), where increased adsorbent weight enhances removal, especially under mildly acidic to neutral conditions. This is attributed to improved interaction between dye molecules and surface functional groups, facilitated by higher surface area.

3.2.3 pH and time effect on adsorption efficiency

The interaction between pH and contact time significantly influences Ni^{2+} adsorption. As shown in Fig. 10 (c), adsorption efficiency increases more rapidly at lower pH values over time. This is due to stronger electrostatic attraction and chelation between Ni^{2+} ions and EDTA-functionalized sites on the adsorbent surface. At higher pH, the formation of $Ni(OH)_2$ complexes reduces the availability of free Ni^{2+} ions, slowing the adsorption process. To support this observation, kinetic data were fitted to three models. The pseudo-first-order plot (Fig. 11(d)) yielded $R^2 = 0.9774$, indicating that the rate depends on the concentration of Ni^{2+}. The pseudo-second-order model (Fig. 11 (e)) showed even stronger correlation ($R^2 = 0.9867$), suggesting chemisorption as the dominant mechanism. The intraparticle diffusion plot (Fig. 11 (f)) revealed a multi-stage process, with initial surface adsorption followed by gradual pore diffusion.

3.2.4 Adsorbent weight and pollutant concentration effect on adsorption efficiency

As shown in Fig. 10 (d), the interaction between adsorbent dose and pollutant concentration significantly affects the adsorption performance. At lower concentrations, increasing the adsorbent dose leads to higher removal efficiency due to the abundance of active sites. However, as the pollutant concentration increases beyond a threshold, the surface of the adsorbent becomes saturated, and the efficiency begins to decline. This is attributed to the limited number of available binding sites, which cannot accommodate excess pollutant molecules. The 3D response surface plot illustrates this saturation effect clearly. A plateau is observed at high concentrations, indicating that the adsorbent has reached its maximum capacity. Optimal conditions are achieved at moderate pollutant concentrations (e.g., 50–75 mg/L) and higher adsorbent doses (e.g., 12–15 mg), where the balance between surface availability and pollutant load is favorable [71].

3.2.5 Time and pollutant concentration effect on adsorption efficiency

Figure 10 (e,k) present the 3D response surface plots illustrating the combined effect of contact time and initial pollutant concentration on removal efficiency for Ni^{2+} and MG, respectively. The plots reveal a nonlinear interaction between these two variables. At lower pollutant concentrations (e.g., < 50 mg/L), the adsorbent surface remains unsaturated, and increasing contact time allows more ions to diffuse and bind to available active sites, resulting in higher removal efficiency. As the initial concentration increases, the number of ions competing for adsorption sites rises sharply. Although the initial uptake may be rapid, the surface approaches saturation more quickly, and the efficiency plateaus or even declines despite extended contact time. This behavior is consistent with the pseudo-second-order kinetic model, where the rate of adsorption slows as equilibrium is approached. The curvature of the response surface confirms that maximum removal occurs under moderate concentrations and sufficient contact time. Beyond a certain concentration threshold, the adsorbent's capacity becomes limiting, and additional time yields diminishing returns. These trends highlight the importance of balancing pollutant load with contact duration to optimize adsorption performance.

3.2.6 Time and adsorbent weight effect on adsorption efficiency

Figure 10 (f,l) illustrate the combined effect of contact time and adsorbent dose on the removal efficiency of Ni^{2+} and MG, respectively. The response surface plots reveal a nonlinear relationship, where increasing both parameters generally enhances adsorption performance.

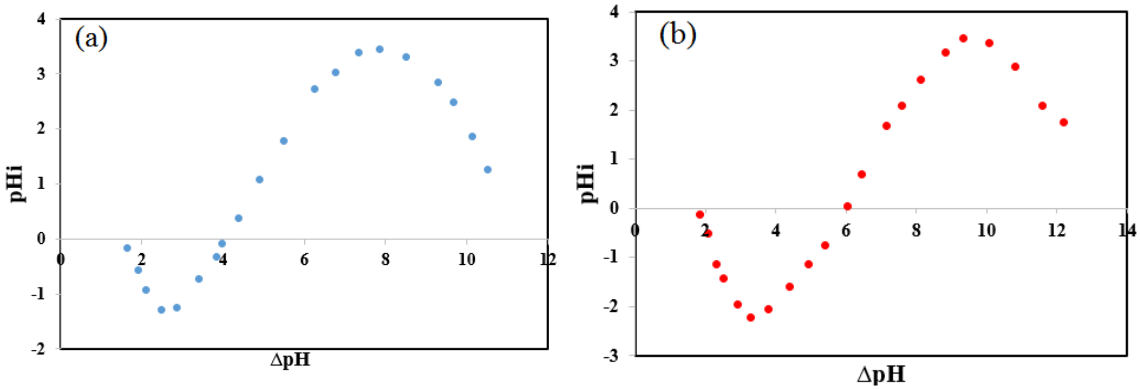
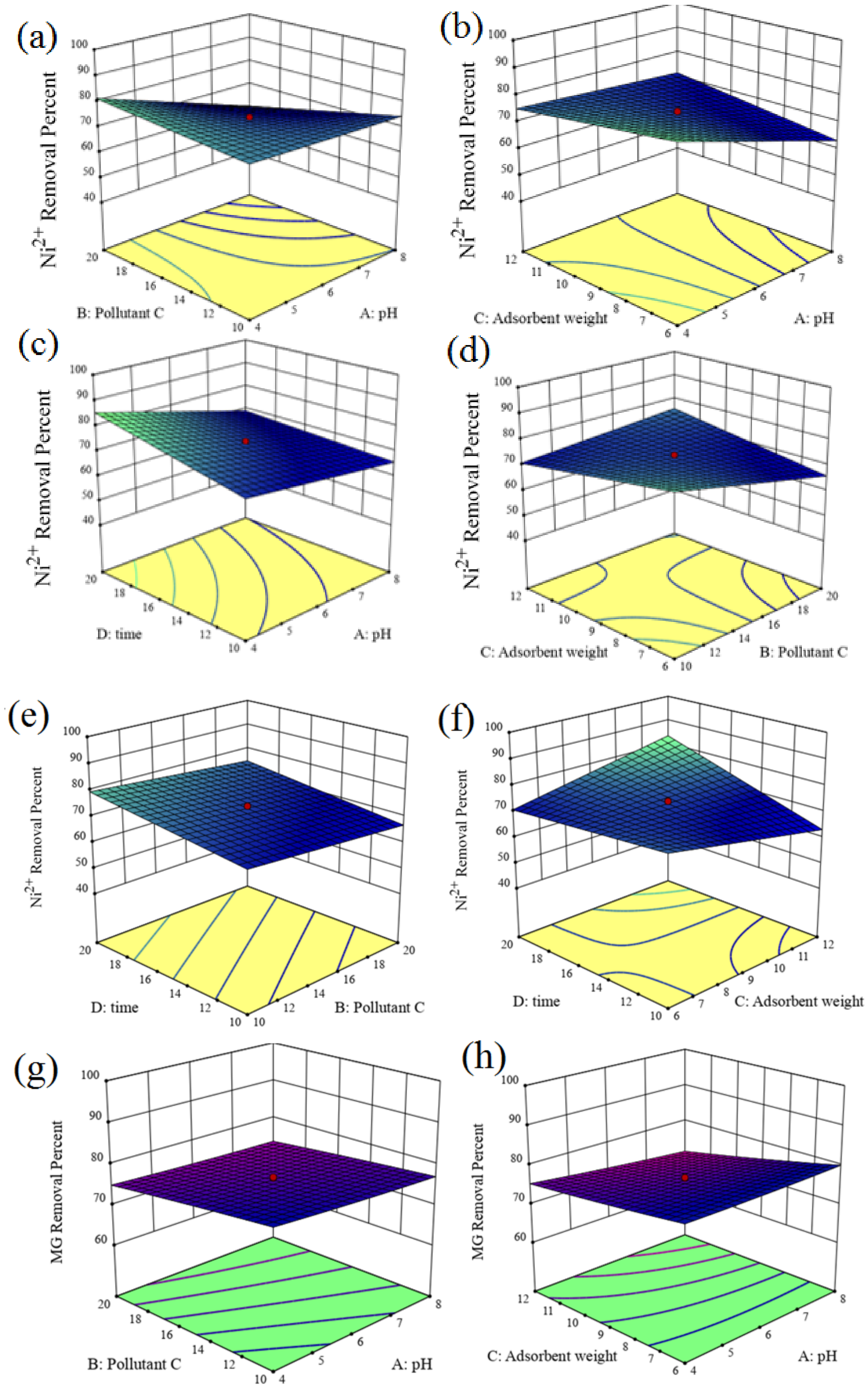


Figure 9. Isoelectric point diagram of EDTA F-MAC for, (a) Ni^{2+} and, (b) MG adsorption.



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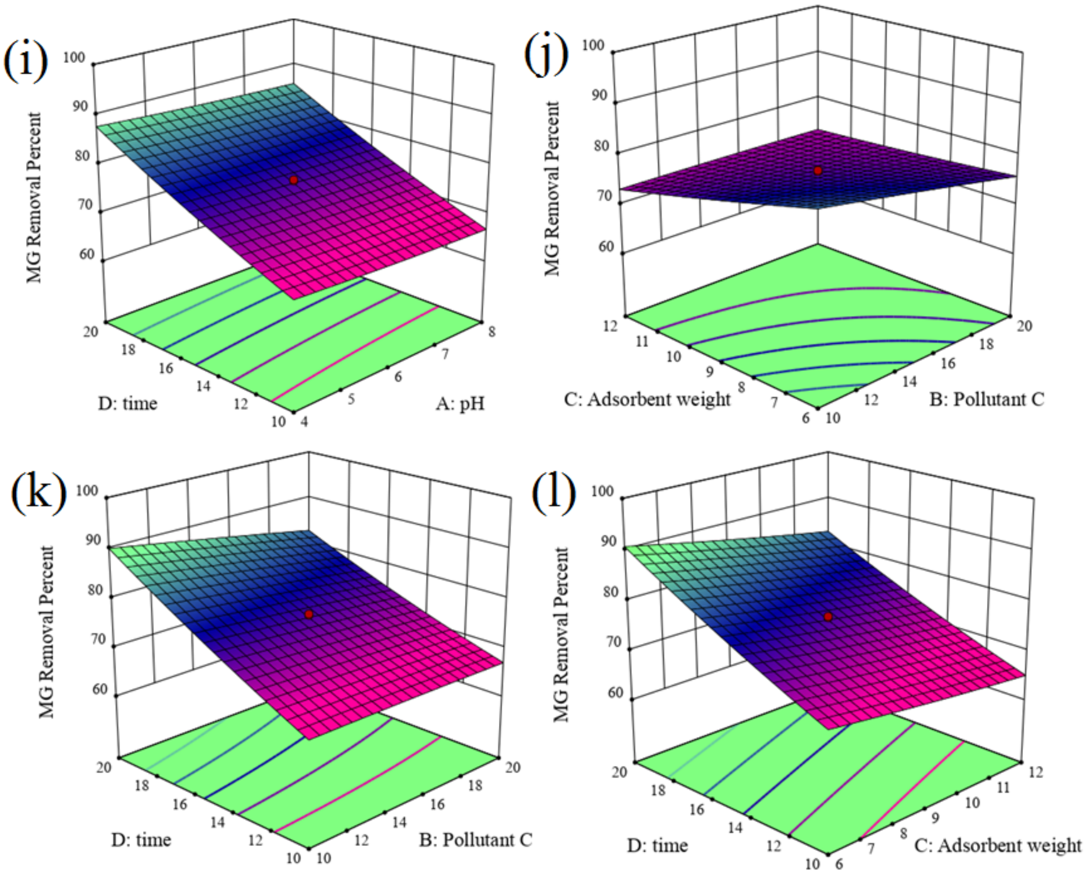

Figure 10. (a-l): The 3D graph for Ni^{2+} and MG removal based on RSM.

A higher adsorbent dose provides more active sites for interaction, while a longer contact time allows sufficient diffusion and binding of pollutant molecules. However, this improvement is pH-dependent and does not occur uniformly across all conditions. At extreme pH values, particularly under alkaline conditions, the formation of hydroxide complexes and changes in surface charge can limit adsorption, even with increased dose and time. The curvature of the plots indicates that maximum efficiency is achieved under mildly acidic to neutral conditions, beyond which the surface approaches saturation and further increases in time or dose yield minimal gains. These trends are consistent with pseudo-second-order kinetics, where rapid initial uptake is followed by a slower approach to equilibrium.

3.3 Adsorption isotherms, reaction kinetics and thermodynamic parameters study

Kinetic data provide valuable insights into the adsorption mechanism, which is essential for optimizing the efficiency of the adsorption process (Figs 11 (a-f)). Understanding the adsorption rate is critical for effective heavy metal removal from water and for refining design parameters. The adsorption rate constant of Ni^{2+} on the EDTA F-MAC adsorbent was evaluated under specific conditions: an adsorbent weight of 16 mg, pH of 4, initial Ni^{2+} concentrations of 5, 10, 15, 20, 25, and 30 mg/L, and a test duration of 1 hour. An experiment was conducted to examine the reaction kinetics, and the collected data were used to construct plots for first-order (Fig. 11 (a)), second-order (Fig. 11 (b)), and intraparticle diffusion models (Fig. 11 (c)). The correlation coefficients were compared, revealing that the adsorption reaction kinetics for EDTA F-MAC followed a first-order model. This indicates that the adsorption rate is directly proportional to the adsorbate concentration in the solution.

The adsorption data were further analysed using various isotherm models, including Langmuir (Fig. 11 (d)), Freundlich (Fig. 11 (e)), and Temkin (Fig. 11 (f)). The results demonstrated the best fit with the Freundlich isotherm, suggesting that the adsorption process on EDTA F-MAC occurs in a multilayer manner over a heterogeneous surface. The value of $1/n$ obtained from the Freundlich model was between 0 and 1, confirming favorable adsorption and indicating surface heterogeneity. Although the Langmuir model was less accurate, it was used to estimate the monolayer adsorption capacity (qmax) for comparison purposes.

For the thermodynamic studies, solutions with Ni2+ concentrations of 15 and 9 mg/L were tested at temperatures of 25 and 45 °C, with contact times ranging from 1 to 25 minutes. Thermodynamic parameters including Gibbs free energy (ΔG), enthalpy change (ΔH), and entropy change (ΔS) were calculated using the following

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Table 5. Final chemical composition of each fertilizer.

ΔT °C	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (kJ/mol)
25	−20.458	4.715	12.43
45	−16.405		

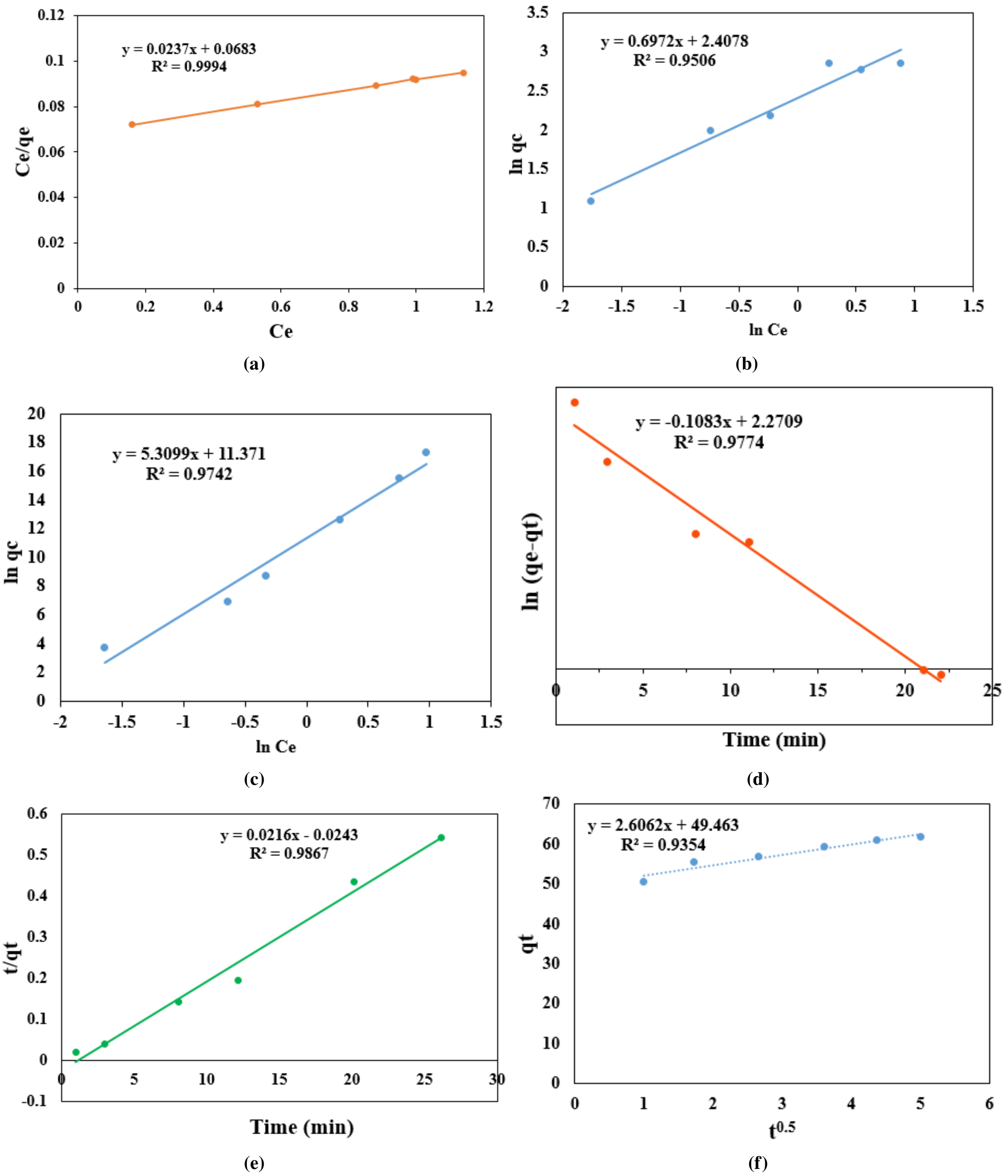
equations [72]:

$$\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \qquad (4)$$

$$\Delta G = \Delta H - T\Delta S \qquad (5)$$

$$\Delta G = -RT \ln K_c \qquad (6)$$

Here, R is the universal gas constant (8.314 J/mol·ºK), T is the absolute temperature in Kelvin, and K_c is the equilibrium constant derived from adsorption data. The negative values of ΔG at both temperatures confirm that the adsorption process is spontaneous. The positive ΔH value (4.715 kJ/mol) indicates that the process is



(a) (b) (c) (d) (e) (f)

Figure 11. (a) First order, (b) Second order, (c) Permeation model kinetics, (d) Langmuir, (e) Freundlich and (f) Temkin isothermal diagram for EDTA F-MAC.

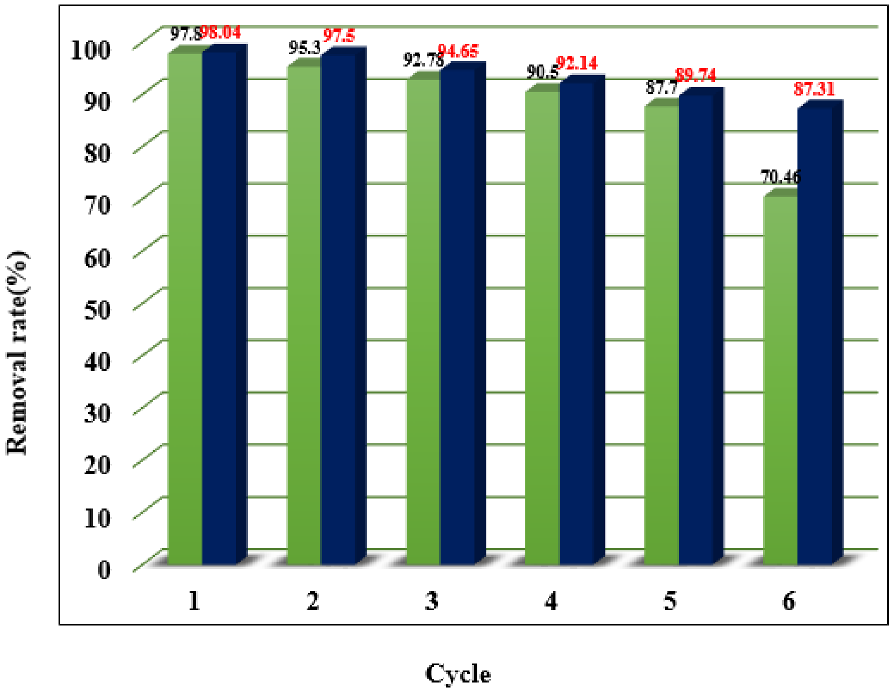


Figure 12. Repeatability of the process, for Ni^{2+} (green) and MG (blue) removal, in 6 consecutive cycles.

endothermic, suggesting strong chemical interactions between Ni^{2+} ions and the EDTA-functionalized adsorbent. Additionally, the positive ΔS value (12.43 J/mol·ºK) reflects increased randomness at the solid–solution interface during adsorption Table 5 [73].

3.4 Reusability of process

To evaluate the reusability of the EDTA F-MAC adsorbent, the adsorption efficiency was tested over six consecutive cycles using a 0.5 M HCl solution under optimal conditions (pH $\approx$ 1, ambient temperature). As shown in Fig. 12, the removal efficiency for Ni^{2+} decreased from 97.8% to 70.46% (a reduction of 27.34%), while MG removal declined from 98.04% to 87.31% (a reduction of 10.73%). These results demonstrate that the adsorbent retains a substantial portion of its performance over multiple cycles, indicating good regeneration capability. The sharper decline in Ni^{2+} efficiency may be attributed to partial detachment or degradation of EDTA functional groups during acid washing, which directly affects metal ion chelation. In contrast, MG adsorption relies more on electrostatic and $\pi-\pi$ interactions, which are less sensitive to regeneration conditions. Overall, the EDTA F-MAC adsorbent exhibits high durability and practical reusability in repeated adsorption–desorption cycles [74, 75].

3.5 Limitations of the study

Despite the promising performance of the developed EDTA-FMAC adsorbent, this study has certain limitations. All experiments were conducted under controlled laboratory conditions, which may not fully reflect the complexity of industrial wastewater systems. Moreover, the long-term structural stability of the adsorbent under continuous-flow operations and in multi-pollutant environments remains to be investigated. Future studies should address these aspects to better assess real-world applicability [76, 77].

4. Conclusion

This research focused on the production of activated carbon from sunflower stems, an agricultural waste material, for the removal of Ni^{2+} and MG from aqueous solutions. Activated carbon was synthesized, magnetized, and subsequently functionalized with EDTA. Structural and functional characterization using XRD, FT-IR, SEM, BET, and VSM confirmed the successful preparation of the EDTA-functionalized magnetic activated carbon. Adsorption experiments were conducted to evaluate the effects of pH, adsorbent dosage, contact time, and pollutant concentration. Under optimal conditions, the removal efficiency reached 94.63% for Ni^{2+} and 97.68% for MG. The experimental results showed strong agreement with the RSM model predictions, validating the reliability of the optimization approach. Despite these promising results, the study has certain limitations. All experiments were performed under controlled laboratory conditions, which may not fully reflect the complexity of industrial wastewater systems. Factors such as long-term structural stability, regeneration cost, and performance under continuous-flow and multi-pollutant environments remain to be investigated. These limitations highlight the need for further validation under real-world conditions. Nevertheless, the EDTA-FMAC adsorbent shows strong potential for industrial water treatment applications, particularly in the selective removal of heavy metals. Future research should focus on pilot-scale implementation, integration into existing treatment systems, and material optimization for enhanced selectivity and durability.

Authors contributions
All authors contributed equally to the conception, design, execution, and writing of this work. All authors read and approved the final manuscript.

Availability of data and materials
The authors declare that the data supporting the findings of this study are available within the paper.

Conflict of interests
The authors assert that they do not have any identifiable conflicting financial interests or personal relationships that might be perceived to influence the work presented in this paper.

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