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Falyouna, Omar
Water and Environmental Engineering Laboratory, Interdisciplinary Graduate School of Engineering Sciences, Kyushu University

Maamoun, Ibrahim
Advanced Science Research Center, Japan Atomic Energy Agency

Mohd Faizul Idham
Water and Environmental Engineering Laboratory, Interdisciplinary Graduate School of Engineering Sciences, Kyushu University

Eljamal, Osama
Water and Environmental Engineering Laboratory, Interdisciplinary Graduate School of Engineering Sciences, Kyushu University

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Effect of water matrix on ligand-assisted adsorption of ciprofloxacin from aqueous solutions by zerovalent iron nanoparticles

Omar Falyouna[1], Ibrahim Maamoun[2], Mohd Faizul Idham[1,3], Osama Eljamal[1*]
[1] Water and Environmental Engineering Laboratory, Interdisciplinary Graduate School of Engineering Sciences, Kyushu University, 6-1 Kasuga-Koen Kasuga, Fukuoka, Japan, 816-8580
[2] Advanced Science Research Center, Japan Atomic Energy Agency, Tokai, Ibaraki 319-1195, Japan
[3] School of Mechanical Engineering, College of Engineering, Universiti Teknologi MARA, Cawangan Terengganu, Malaysia
*Corresponding author email: osama-eljamal@kyudai.jp

Abstract: *The adsorption of the antibiotic ciprofloxacin (CIP) from natural water and wastewater could be challenging due to the presence of competing compounds and elements. This study illustrates the negative influence of ionic strength, coexisting ions, and natural organic matter on the adsorption of CIP by oxalate-modified zerovalent iron nanoparticles (*Fe^0*/oxalate). The results showed that the ionic strength of aqueous solution slightly decreased CIP removal efficiency from 95.43% to 85.23% after increasing sodium chloride concentration to 100 mM. On the contrary, the competence of (*Fe^0*/oxalate) nanoparticles in adsorbing CIP slightly dropped from 95.43% to 94.42% due to the increase of humic acid from 0 to 40 mg* L^{-1}*. Also, the outcomes conclude that the inhibitory effect of 10 mM of coexisting ions was ranked as follows:* $K^+ < CO_3^{2-} < Ca^{2+} < SO_4^{2-} < NO_3^- < Mg^{2+}$.

Keywords: Ciprofloxacin**;** Zerovalent iron nanoparticles (Fe^0); Oxalate; Natural organic matter (NOM), Co-exiting ions.

1. INTRODUCTION

Antibiotics are a class of synthetic and natural organic substances developed to treat various infectious diseases [1,2]. Salvarsan was the first developed antibiotic in 1910 [3]. However, after the discovery of penicillin by Sir Alexander Fleming (1881-1955) in 1928, the golden era of antibiotics had started with the development of a broad spectrum of novel antibiotics [3,4]. The utilization of antibiotics extended the lifespan of humans by 23 years and dramatically changed the features of modern medicine [3]. For example, the application of antibiotics not only helped in treating bacterial infections but also made many complicated medical surgeries possible such as open-heart surgeries, organ transplants, cancer treatment, etc. [3,5]. Residues of antibiotics are released to the environment through the effluents of domestic, industrial, and hospital wastewater treatment plants, landfill sites, various veterinary applications (e.g., aquaculture, poultry and livestock farms, etc.), and so forth [6,7]. Despite the great benefits in the human and veterinary medicine, the persisting and bioaccumulation of antibiotics in the environment especially the limited water resources are suspected of causing serious health and ecological issues. For an instance, the frequent occurrence of antibiotics in water promotes the growth of antimicrobial resistant bacteria. This kind of bacteria cause incurable and deadly diseases [8]. According to the UK Government-commissioned O'Neill report, 70000 people are dying annually due to the antimicrobial resistant bacteria and it is expected that this figure is going to increase to 10 million people by 2050 [9]. Ciprofloxacin (CIP) is a second-generation fluoroquinolone antimicrobial agent used to treat many bacterial infections, for example, infections of gastrointestinal tract, respiratory system, urinary tract, bones, joins, and so on [10]. CIP is one of the most frequently detected antibiotics in several water systems such as surface water (i.e., rivers, lakes, bonds, etc.), groundwater, wastewater, etc. [4,11]. Thus, an effective treatment technology is necessary to reduce CIP concentrations in water and to protect the public from the adverse health effects of CIP and other antibiotics.

Zerovalent iron nanoparticles (Fe^0) are one of the most employed nanomaterials in the remediation of recalcitrant contaminants from water [12–14]. They are featured by distinctive core-shell form, small particle size (<100 nm), high surface area, high reactivity towards contaminants, nontoxicity, and cost-effectiveness [15,16]. Fe^0 nanoparticles have many limitations in water and wastewater treatment applications such as rapid passivation, particle agglomeration, poor mobility in porous media, and low oxidants yields [17–19]. Many modifications were adopted to improve the performance of Fe^0 nanoparticles in field-scale applications, for example, incorporation of noble metals (e.g., Ni, Cu, Ag, Pd, etc.), supportation by various materials (e.g., carbon nanotubes, graphene oxide, biochar, zeolite, clays, etc.), surface stabilization (e.g., polymers, surfactants, etc.), emulsification, oxidant addition (i.e., hydrogen peroxide), and organic ligands addition (e.g., oxalate, etc.) [7,20,21]. Our previous article demonstrated that the adding 0.3 mM of the organic ligan oxalate to 0.3 g L^{-1} of Fe^0 nanoparticles enhanced the adsorption of CIP from 45.04% (Fe^0) to 95.74% (Fe^0/oxalate) [4]. These results were obtained under ideal conditions, where CIP was adsorbed from deionized water. However, the actual wastewaters and natural waters have many foreign substances and elements that obstruct the remediation of CIP in real contamination scenarios. Therefore, this study aims to study the effect of ionic strength, coexisting ions, and natural organic matter on the elimination of CIP by (Fe^0/oxalate) nanoparticles to ensure their suitability for field-scale applications.

2. MATERIALS AND METHODS

2.1 Materials

Ciprofloxacin hydrochloride monohydrate ($C_{17}H_{18}FN_3O_3.HCl.H_2O$) was purchased from Tokyo Chemical Industry CO., LTD (Japan). In addition, ferric chloride hexahydrate ($FeCl_3.6H_2O$) and sodium borohydride ($NaBH_4$) were obtained from JUNSEI (Japan) and Sigma Aldrich (USA), respectively. Moreover, sodium sulfate (Na_2SO_4), potassium chloride (KCl), sodium carbonate (Na_2CO_3), sodium chloride (NaCl), and sodium oxalate ($Na_2C_2O_4$) were acquired from FUJIFILM Wako Pure Chemicals (Japan). Also, calcium chloride dihydrate ($CaCl_2.2H_2O$) and sodium nitrate ($NaNO_3$) were supplied by JUNSEI (Japan). Furthermore, humic acid was obtained from Sigma Aldrich (USA). The solutions in this research were prepared by using deionized water (DIW).

2.2 Synthesis of zerovalent iron nanoparticles (Fe^0)

Fe^0 nanoparticles were produced in the laboratory by the chemical reduction technology, where the ferric ions (Fe^{3+}) were reduced to (Fe^0) via a reducing agent such as sodium borohydride, as shown in Fig.1 [22,23]. In detail, to produce one gram of Fe^0 nanoparticles, 0.093 M of ferric (Fe^{3+}) solution and 0.58M of borohydride (BH_4^-) solution were prepared as follows: 5 grams of $FeCl_3.6H_2O$ were mixed with 200 ml of DIW and 2.2 grams of NaBH4 were mixed with 100 ml of DIW. The solutions were magnetically mixed for 15 min to ensure the complete dissolution of the added chemicals. Also, it is important to mention that DIW was purged with pure nitrogen gas (N_2) for 10 min before the preparation of ferric and borohydride solutions to minimize oxidation of Fe^0 nanoparticles. After the preparation of the two solutions, the ferric solution was decanted into a four-neck flask as demonstrated in Fig.1 and the borohydride solution was added to the ferric solution by using a peristaltic pump with a flow rate of 20 ml min^{-1}. After the addition of the borohydride solution, the mixture of the solutions was agitated under N_2 atmosphere via a mechanical mixer at 400 rpm and under a temperature of 30 °C for 5 min to guarantee the complete reduction of Fe^{3+} ions to Fe^0. The resulted Fe^0 nanoparticles were then collected by using a vacuum filtration system, as illustrated in Fig.1.

2.3 Batch experiments

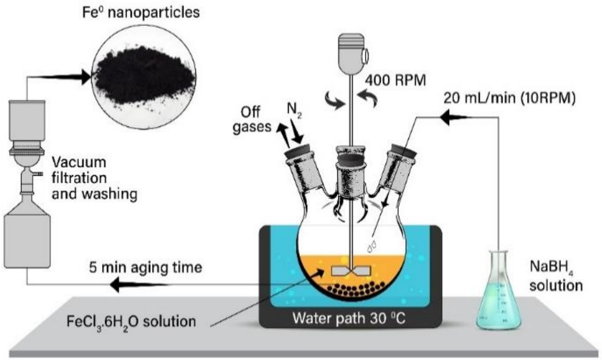

Fig. 1. Schematic diagram for the synthesis of Fe0 nanoparticles.

100 mg L^{-1} CIP solution was prepared by dissolving 111 mg of ciprofloxacin hydrochloride monohydrate in 1000 ml of DIW. Batch experiments were conducted to evaluate the performance of (Fe^0/oxalate) nanoparticles in removing CIP under the influence of ionic strength, natural organic matter, and coexisting ions. Our previous article indicated that the optimum removal conditions of CIP by (Fe^0/oxalate) nanoparticles were as follows: [oxalate] = 0.3 mM, [Fe^0] = 0.3 g L^{-1}, [CIP] = 100 mg L^{-1}, initial pH = 7, temperature = 25 °C, treatment time = 30 min and mixing speed = 500 rpm [4]. These conditions were applied in the batch experiments to study the effect of ionic strength, coexisting ions, and natural organic matters on the competence of (Fe^0/oxalate) nanoparticles in remediating CIP-contaminated water. The reaction between (Fe^0/oxalate) nanoparticles and CIP started after placing the flask on a magnetic stirrer at 500 rpm for 30 min. At the end of the treatment process, 2 ml liquid sample was taken by a plastic syringe and filtered by 0.45 µm filter and kept in 2 ml sampling tube for further analysis.

2.4 Analytical instruments

The residual concentration of CIP after the treatment process was measured by UV-vis spectrophotometer (UV-1280, SHIMADZU, Japan) at a wavelength of 275 nm [24]. The UV-1280 was calibrated using 8 standard solutions (i.e., 0.25, 0.5, 0.75, 1, 2, 3, 4, and 5 mg L^{-1}) before using it to measure CIP concentration. The calibration curve of the UV-1280 is as follows:

$$Y = 8.911X \tag{1}$$

Y and X symbolize the concentration of CIP (mg L^{-1}) and the absorbance value of the UV-1280 at 275 nm.

2.5 Evaluation of the performance of (Fe^0/oxalate) nanoparticles

The competence of (Fe^0/oxalate) nanoparticles was assessed by calculating the final removal efficiency (RE) using the following equation [25,26]:

$$\text{RE}\ (\%) = \frac{(C_i - C_f)}{C_i} \times 100\% \tag{2}$$

C_i and C_f represent CIP initial and final concentrations (mg L^{-1}), respectively.

3. RESULTS AND DISCUSSION

3.1 Effect of ionic strength

Pharmaceutical and industrial wastewater usually contain high concentrations of salts [27]. Hence, to predict the competence of (Fe^0/oxalate) nanoparticles in removing CIP from wastewater, the impact of ionic strength, represented by sodium chloride (NaCl), was investigated by increasing the concentration of NaCl from 0 to 100 m M, as shown in Fig.2.

Fig.2 demonstrates that the influence of the ionic strength on the removal of CIP by (Fe^0/oxalate) nanoparticles was neglectable when the ionic strength was less than 50 mM. On the other hand, the removal efficiency of CIP declined from 92.75% to 85.23% after increasing the ionic strength from 50 to 100 mM. The presence of excessive concentrations of NaCl (e.g., 100 m M) in the aqueous solution may increase the competition with CIP molecules on the adsorption locations of (Fe^0/oxalate) nanoparticles and decrease the overall removal efficiency.

3.2 Effect of natural organic matter (NOM)

Natural waters and wastewaters also comprise natural organic matter such as humic acid, fulvic acid, etc. [28]. These compounds have many functional groups, for example, alcoholic, carboxyl, hydroxyl, and phenolic groups. These groups could interact with (Fe^0/oxalate) nanoparticles and obstruct the removal of CIP from water. Thus, the impact of natural organic matter (NOM) on the effectiveness of (Fe^0/oxalate) nanoparticles was examined by increasing the concentration of humic acid, as a representative for the natural organic matter, from 0 to 40 mg L^{-1}, as illustrated in Fig.3.

Fig.3 manifests that the remediation efficiency of 100 mg L^{-1} of CIP by (Fe^0/oxalate) nanoparticles was trivially decreased from 95.43% to 94.42% after increasing the concentration of humic acid from 0 to 40 mg L^{-1}. These results proved that humic acid didn't intervene or compete with the molecules of CIP on the adsorption sites of (Fe^0/oxalate) nanoparticles. Hence, it can be anticipated that (Fe^0/oxalate) nanoparticles will effectively eliminate CIP from actual contaminated water regardless of the concentration of natural organic matter in the water.

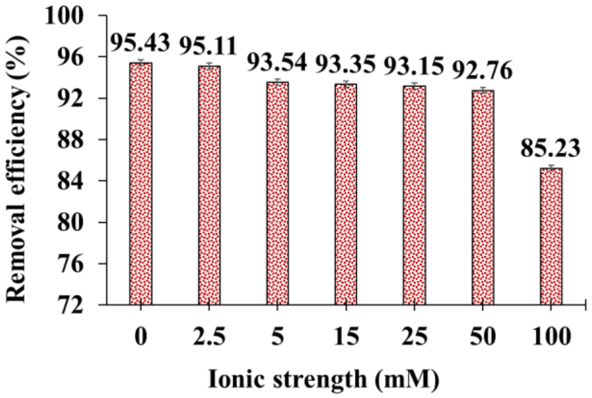

Fig. 2. Effect of ionic strength on the efficacy of (Fe^0/oxalate) nanoparticles in removing CIP from water.

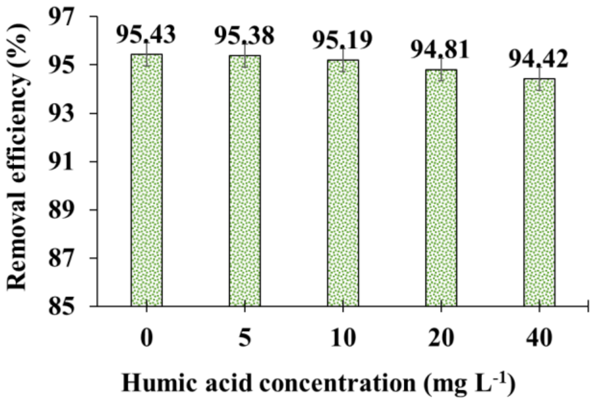

Fig. 3. Effect of humic acid on the efficacy of (Fe^0/oxalate) nanoparticles in removing CIP from water.

3.3 Effect of coexisting ions

The composition of domestic, industrial, or hospital wastewater is complex and includes a variety of elements besides Na^+ and Cl^- [29]. Some of these elements can strongly interfere with the adsorption of CIP by (Fe^0/oxalate) nanoparticle and negatively affect the treatment efficiency. Therefore, it is essential to understand the influence of coexisting ions on the performance of (Fe^0/oxalate) to grantee the successful remediation of CIP from different types of wastewaters.

3.3.1 Effect of coexisting cations

The impact of coexisting cations was explored by varying the concentrations of potassium (K^+), calcium (Ca^{2+}), and magnesium (Mg^{2+}) in the aqueous solution, as elucidate in Fig.4. The concentration of each cation was changed within a specific range based on the hindrance effect. The concentration range of each cation was as follows: $0 < K^+ < 50$ mM, $0 < Ca^{2+} < 30$ mM, and $0 < Mg^{2+} < 10$ mM. Fig.4.a illustrates that the removal efficiency of CIP by (Fe^0/oxalate) nanoparticles was slightly decreased from 95.43% to 91.82% due to the increase of the potassium concentration from 0 to 50 mM. On the other hand, it can be seen from Fig.4.b, and Fig.4.c that the divalent cations are strongly competing with the molecules of CIP on the adsorption sites of (Fe^0/oxalate) nanoparticles. Fig.4.b indicates that the removal efficiency of CIP notably dropped from 95.43% to 57.31% as a result of increasing calcium concentration from 0 to 30 mM. Also, Fig.4.c exhibits that competence of (Fe^0/oxalate) nanoparticles severely deteriorated from 95.43% to 37.89% because of increasing magnesium concentration from 0 to 10 mM. These results confirm that at a concentration of 10 mM, the inhibitory effect of coexisting cations were in the following order: $K^+ < Ca^{2+} < Mg^{2+}$.

3.3.2 Effect of coexisting anions

The influence of coexisting anions on the performance of (Fe^0/oxalate) nanoparticles in eliminating CIP from water was examined by changing the concentration of nitrate (NO_3^-), sulphate (SO_4^{2-}), and carbonate (CO_3^{2-}) ions in the water, as displayed in Fig.4. The concentration range for the coexisting anions was as follows: $0 < NO_3^- < 30$ mM, $0 < SO_4^{2-} < 10$ mM, and $0 < CO_3^{2-} < 10$ mM. The removal efficiency of CIP by (Fe^0/oxalate) nanoparticles considerably declined from 95.43% to 58.81% after increasing the concentration of nitrate from 0 to 30 mM (Fig.4.d). This deficiency could be attributed to the consumption of the electrons released from the core of (Fe^0/oxalate) nanoparticles to reduce and eliminate nitrate from the water. The excessive release of electrons from the core of (Fe^0/oxalate) nanoparticle weakens their adsorption capacity towards CIP. Similarly, Fig.4.e and Fig.4.f confirm that the presence of sulphate and carbonate ions in the aqueous solution with a concentration of 10 mM diminished the competence of (Fe^0/oxalate) in removing CIP from water by 19.83% and 15.02%, respectively. From these findings, it can be anticipated that the existence of high concentration of nitrate, sulphate and carbonate ions in the contaminated water could substantially deteriorate the efficiency of (Fe^0/oxalate) nanoparticles in remediating CIP-contaminated water. In conclusion, at a concentration of 10 mM, the adverse impact of coexisting anions can be ranked as follows: $CO_3^{2-} < SO_4^{2-} < NO_3^-$.

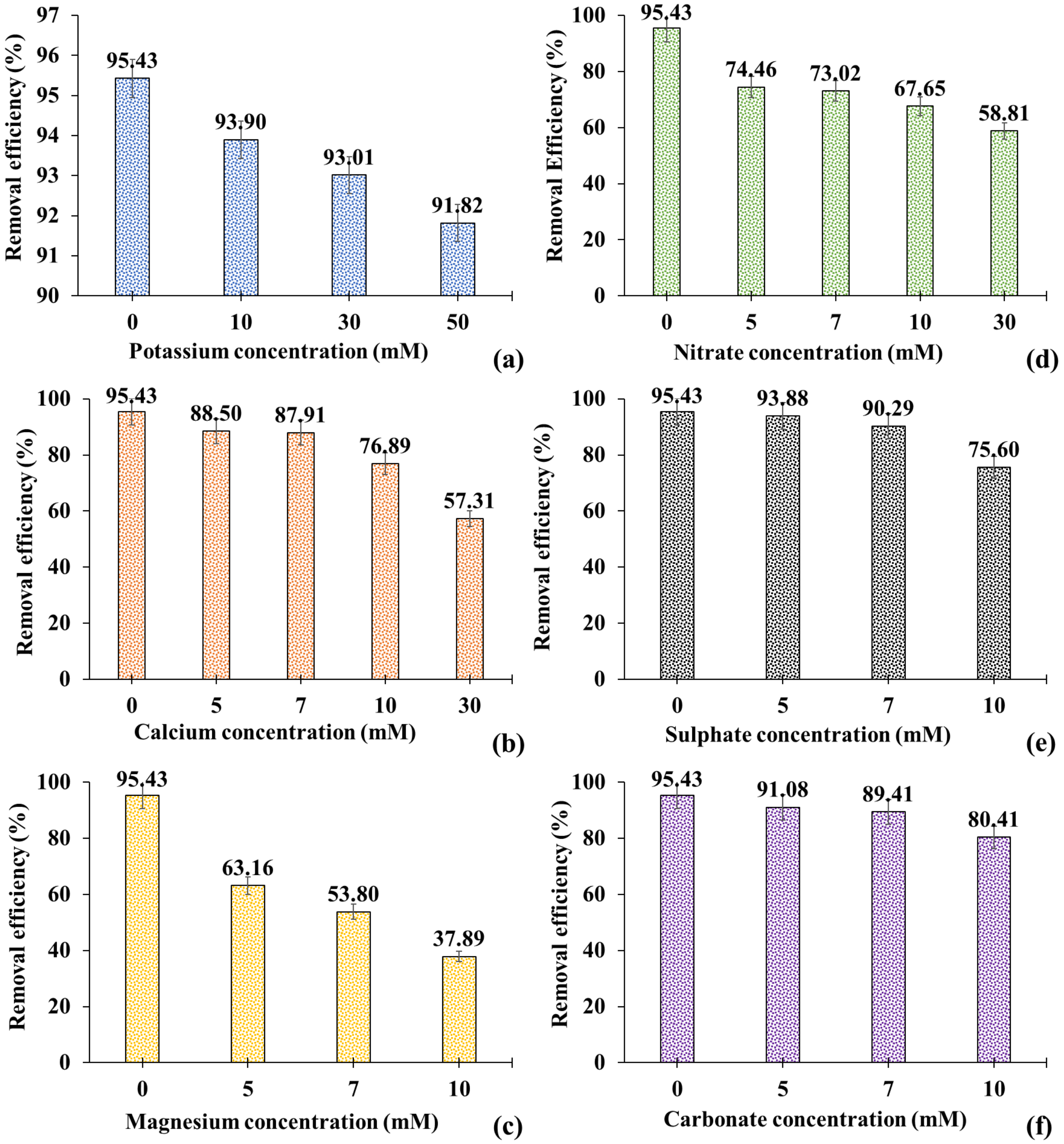

Fig. 4. Effect of coexisting on the efficacy of (Fe^0/oxalate) nanoparticles in removing CIP from water: (a) potassium, (b) calcium, (c) magnesium, (d) nitrate, (e) sulphate, and (f) carbonate.

3.3.3 Effect of coexisting anions

The influence of coexisting anions on the performance of (Fe^0/oxalate) nanoparticles in eliminating CIP from water was examined by changing the concentration of nitrate (NO_3^-), sulphate (SO_4^{2-}), and carbonate (CO_3^{2-}) ions in the water, as displayed in Fig.4. The concentration range for the coexisting anions was as follows: $0 < NO_3^- < 30$ mM, $0 < SO_4^{2-} < 10$ mM, and $0 < CO_3^{2-} <10$ mM. The removal efficiency of CIP by (Fe^0/oxalate) nanoparticles considerably declined from 95.43% to 58.81% after increasing the concentration of nitrate from 0 to 30 mM (Fig.4.d). This deficiency could be attributed to the consumption of the electrons released from the core of (Fe^0/oxalate) nanoparticles to reduce and eliminate nitrate from the water. The excessive release of electrons from the core of (Fe^0/oxalate) nanoparticle weakens their adsorption capacity towards CIP. Similarly, Fig.4.e and Fig.4.f confirm that the presence of sulphate and carbonate ions in the aqueous solution with a concentration of 10 mM diminished the competence of (Fe^0/oxalate) in removing CIP from water by 19.83% and 15.02%, respectively. From these findings, it can be anticipated that the existence of high concentration of nitrate, sulphate and carbonate ions in the contaminated water could substantially deteriorate the efficiency of (Fe^0/oxalate) nanoparticles in remediating CIP-contaminated water. In conclusion, at a concentration of 10 mM, the adverse impact of coexisting anions can be ranked as follows: $CO_3^{2-} < SO_4^{2-} < NO_3^-$.

4. CONCLUSIONS

This paper discusses the influence of ionic strength, natural organic matter, and coexisting ions on the removal efficiency of CIP from water by (Fe^0/oxalate) nanoparticles. The outcomes of this research proved that the ionic strength of the water, within an NaCl concentration range of 0 – 100 mM, had a trivial effect on the competence of (Fe^0/oxalate) nanoparticles. In addition, the performance of (Fe^0/oxalate) nanoparticles wasn't affected by the presence of humic acid with a concentration ranged from 0 to 40 mg L^{-1}. In contrast, the existence of coexisting cations (K^+, Ca^{2+}, and Mg^{2+}) and anions (NO_3^-, SO_4^{2-}, and CO_3^{2-}) decreased the effectiveness of (Fe^0/oxalate) nanoparticles in remediating CIP-contaminated water. At a concentration of 10 mM, the coexisting ions could hinder the removal of CIP in the following order: $K^+ < CO_3^{2-} < Ca^{2+} < SO_4^{2-} < NO_3^- < Mg^{2+}$.

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