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Research Paper

Removal of Ni(II) and Cr(VI) Ions From Electroplating Wastewater Using Ferrous Sulfate

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ABSTRACT

Background: In this study, the ferrous sulfate was used to remove Cr(VI) and Ni(II) ions from a real Ni-Cr plating industry wastewater.

Methods: This is an applied study. The type of sampling was composite by three 5 L containers. Wastewater chemical and physical characteristics were measured in accordance with the standard methods. The concentration of un-removed metal ions was estimated by ICP-OES. The Jar test was used to carry out the chemical coagulation experiment. The results indicated that the rate of removal depended on the pH of 1.5-11, the diverse contact time of 30-120 min, and the coagulant measurement ranging from 0.5-2 g/L.

Results: The optimum removal of Cr(VI) ions was observed at a pH of 7 up to 83.88% in 0.5 g/L of coagulant for 40 min. While the optimum removal of Ni(II) ions was found at a pH of 5 up to 56.33 % in 0.5 g/L of coagulant for 40 min. The scanning electron microscopy results revealed the difference between the coagulant surface while coagulation. Also, the Energy-Dispersive X-ray spectroscopy (EDX) analysis showed the presence of Cr(VI) and Ni(II) ions on the surface of the ferrous sulfate after coagulation.

Conclusion: Ultimately, the results indicated that ferrous sulfate could be an operative chemical coagulant to remove Cr(VI) and Ni(II) ions from the plating industry wastewater, especially for Cr(VI) particles. The results of this study can be a useful guide to the real plating industry wastewater treatment operators.

1. Introduction

Because of unsafe and long-lasting impacts on human and ecological safety, heavy metals, which are typically created by modern exercises and technology development are normally considered as the toxins causing one of the most important concerns. Heavy metals are the primary cause of hazards to humans, animals, and plants [1]. They cause diseases, such as kidney, liver, and lung disorders, brain diseases or memory decline, growth deficiency, cancer, and in extraordinary cases, demise [2]. The activities and materials, like jewelry, leather, battery, painting oil refining,

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mineral preparing factories, tannery, and ceramic manufacturing produce wastewater heavy metal particles [3]. These materials are poisonous and chronic exposure to them can cause mutagenesis and carcinogenesis [4]. Additionally, they can represent a danger to the existence cycle because of their poisonousness, aggregation, non-biodegradable nature, and persistence [5]. Heavy metals can harmfully affect the primary function of organs, like the liver, nerves, kidney, and bones, and also necessary enzymes by restraining the practical compounds [6]. Nickel (Ni) and Chromium (Cr) are two of the most important naturally occurring metals in the electroplating cycle [7]. There are elevated levels of heavy metals, especially Cr and Ni in plating and metalworking industry wastewater (1) Only 60-70% of the metals are utilized in plating measures [8]. Untreated effluents from these manufacturing processes harm the environment [7]. The World Health Organization (WHO) recommends that the toxic limits of Cr(VI) and Ni in wastewater are 0.05 mg/L and 0.2 mg/L, respectively [8]. Ni is present in the wastewater of different production lines, for example, Ni coating and metal completing, steel assembling, jewelry making, mining, capacitor sequential construction systems, and transfer heat systems. This heavy metal can cause unfavorably susceptible responses and persistent asthma, heart and blood disorders, dermatitis, and cancer [8, 9]. Modern effluents from Ni plating businesses contain high content of Ni ions and are of noteworthy concern because they are non-biodegradable in the natural food chains and profoundly poisonous [10]. Ni compounds, such as nickel sulfide, are even suspected to cause cancer. Water containing high levels of this metal is not drinkable and can lead to pulmonary embolism, kidney failure, digestive system damage, and dermatitis, and skin rashes [8, 9]. Similarly, Cr is a harmful and cancer-causing toxin for people [11, 12]. Cr and its compounds are found in metal scratching, chromium plating, metal processing, steel industries, ceramic, wood preservatives, agricultural runoff, tanning, fabric coloring, leather, and cement. It can cause headaches, failure in stomach function, ulcers, loose bowels, nausea, and spewing [8]. Thus, there is a need to treat wastewater before it is released into different water sources. Nonetheless, heavy metals could be taken out from the wastewater using diverse coagulation processes, and lately, coagulation as a basic and reasonable strategy is successfully utilized to eliminate heavy metals from industrial wastewaters. It has additionally been utilized in combination with precipitation methods to treat metal-contaminated solutions. The coagulant destabilizes the suspended pollutes so that the particles connect and agglomerate leading to shaping flocs that drop out of the solution by sedimentation [10]. Coagulants are added to the wastewater to overcome the repulsive forces and increase the van der Waals forces of attraction in the system. Nontoxic, low cost, meeting health authority standards, function at a wide range of pH, and great coagulation impacts are determination standards for coagulants [8].

It has been indicated that probably the best choice to treat polluted wastewaters is utilizing inorganic coagulants, such as aluminum sulfate, ferric chloride, calcium oxide, ferrous sulfate, and Poly Aluminum Chloride (PAC) [8]. Iron coagulants are accessible as ferric sulfate, ferric chloride, and ferrous sulfate. Rahbar et al. in their study showed that ferrous sulfate was more suitable than different coagulations in eliminating the Biochemical Oxygen Demand (BOD) and color and might be considered a superior coagulant than the other hydrolyzing metallic salts [13]. Ukiwel et al. regarding the capacity of ferrous sulfate, aluminum sulfate octadecahydrate, ammonium aluminum sulfate dodecahydrate, and ammonium ferrous sulfate in eliminating turbidity showed that ferrous sulfate was the best coagulant [14]. Omar et al. assessed ferrous sulfate, ferric chloride, and alum and showed that ferrous sulfate was the most economic treatment for painting industry wastewater [15]. Comparing the treatment efficiency of polyaluminium chloride, ferrous sulfate, and ferric chloride in eliminating Chemical Oxygen Demand (COD) and color, it was found that ferrous sulfate created the least sludge for synthetic wastewaters [16].

Moreover, ferrous sulfate is more effective in comparison with alum in removing fluoride and phosphate [17]. Likewise, inorganic salts, such as ferrous sulfate can be successfully applied for essential coagulation [18]. They are highly utilized perhaps because they are suitable for the natural ecological Fe(II) cycle and also are proper for the treatment of polluted groundwater due to their low cost, ease of use, effectiveness, and independence on pH changes in most water types. Fe salts are preferred over Al salts in water and wastewater treatment [14]. Among ferrous sulfate, alum, and aluminum chloride, ferrous sulfate is the best coagulant for COD removal. It has been suggested as a coagulant with a minimum sludge creation in all the cases [19] and has the capacity to eliminate contamination at different temperatures [14]. Also, Samadi et al. in their study found that the best coagulant for COD removal was ferrous sulfate [3]. The current examination was done to assess the capability of ferrous sulfate to remove two heavy metals of (Cr(VI) and Ni(II)) from plating wastewater and explore their potential as a substitute for commercial coagulants. In this study, a real wastewater sample (not a synthetic sample) was used. Also, the experiments were performed to obtain the optimal conditions. Therefore, the research results are more accurate and efficient for use in industrial wastewater treatment.

2. Materials and Methods

Industrial effluent sample

For this study, articles were searched in international databases, like Google Scholar, ScienceDirect as well as the Persian databases of SID and Google Scholar. Articles related to the objectives of the study were identified using keywords, such as Ni(II), Cr(VI), ferrous sulfate, plating wastewater, and coagulant. After the search, sampling was performed from a plating industry in the Ghalemir industrial town, Baharestan, Tehran, Iran. Three 5 L plastic containers previously washed and rinsed with distilled water were used to collect the sample. Three samples were separately collected from the industrial effluent discharge point of the plating. The three samples were then mixed together to form a homogeneous mixture in a 20 L plastic container, previously washed and rinsed with distilled water. The mixture (sample) in the 20 L plastic container was then labeled and transferred to the Laboratory. The samples were kept at 4 °C. A multi-parameter model HQ40 company HACH was used to measure the pH and Electrical Conductivity (EC). Total Dissolved Solids (TDS), COD, and BOD_5 parameters were calculated in accordance with the standard procedures [14]. The physical and chemical attributes of the gathered raw wastewater are recorded in Tables 1 and 2. In this investigation, deionized water was used in all the experiments to prepare working solutions [15]. The study was performed on a laboratory scale using the jar test (model 7790-402).

Based on the results, Ni-Cr plating wastewater has a high concentration of Ni and Cr (Table 1). Also, the pH of the sample is acidic (Table 2). It is because of the utilization of acids, like HCL, H_2SO_4, H_3BO_3, and H_2CrO_4 in plating processes. Figure 1 shows a schematic diagram of the study process.

The apparatus used

- pH – multi-parameter model HQ40 company HACH
- Magnetic stirrer
- Sedimentation beaker
- Stopwatch
- 20 liters of a white gallon
- 5 liters of a gallon
- Beaker (500 ml)
- Whatman filter paper
- Electronic weighing balance
- Thermo iCAP 6000 (ICP-OES)
- Jar test apparatus (model 7790-402)

The materials used

- Buffer solutions of pH
- Conc, H_2SO_4, and NaOH (MERCH Germany)
- Deionized water
- Clean tap water/Base water
- Ferrous sulfate (MERCH Germany)

Chemical analysis

The corresponding gel-like hydroxides and some positively charged mononuclear and polynuclear species are shaped during the hydrolysis of ferrous sulfate in the coagulation process. The mix of positively charged particles with negatively charged particles in the wastewater by the charge neutralization mechanism and the sedimentation of these hydroxides and complexes hydroxides can precipitate out uncharged/charged colloidal of the wastewater [20] The effectiveness of the coagulation process profoundly depends on the optimum coagulant dosage, solution pH, and sedimentation time [17].

Laboratory grade ferrous sulfate ($FeSO_4.7H_2O$) was used as a coagulant. For adjusting the pH of the medium, 0.1 N NaOH or H_2SO_4 was used. The other chemical additives used in the preparation of the synthetic wastewater were of analytical grade. Chemical coagulation experiments were carried out using a jar testing technique.

Accordingly, several important parameters concerning the rate of removal were estimated, including contact time (1/2, 1, 1.5, and 2 h), coagulant dosage (0.5, 1, 1.5, and 2 g/L), and different pH values (1.5, 3, 5, 7, 9, and 11). The temperature was also controlled during the experiments (25 °C).

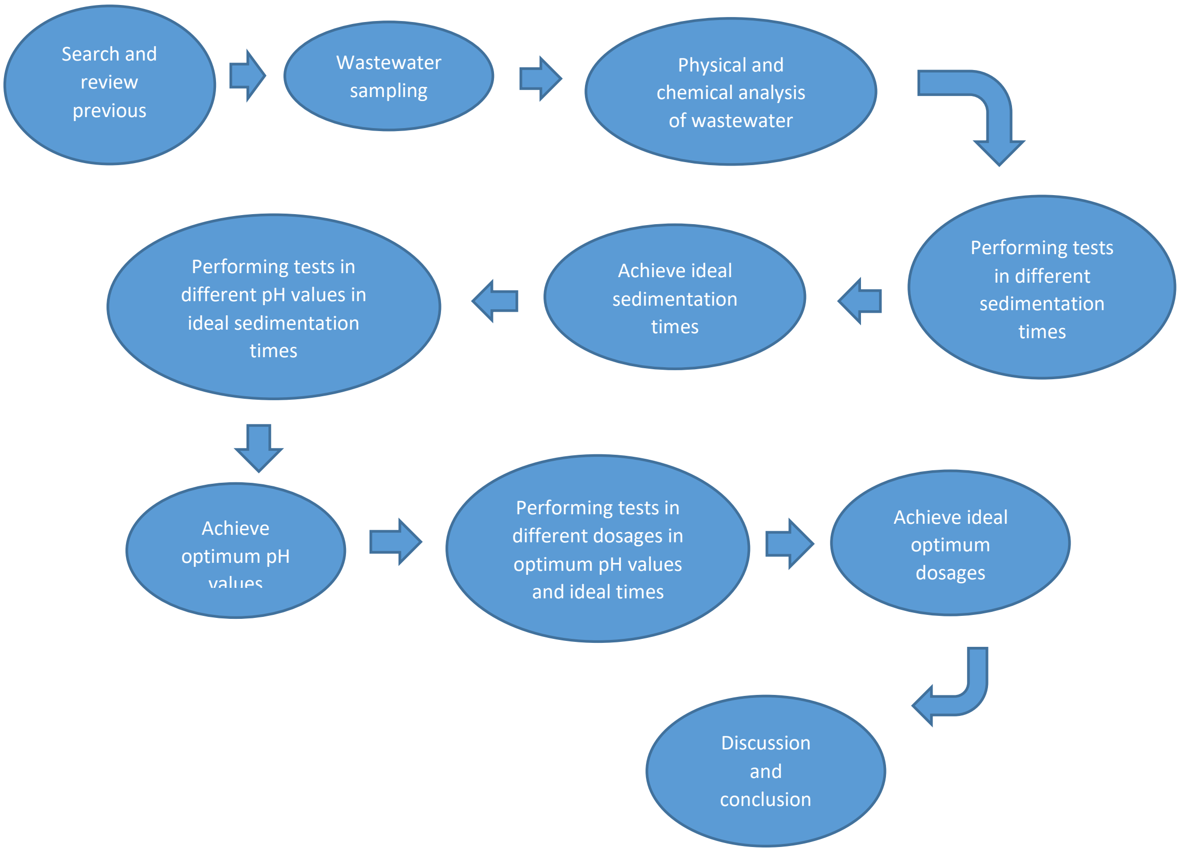


Figure 1. Schematic diagram of the study proces

For examination, 100 ml of the plating wastewater and the ideal measure of coagulant were placed into a 500 ml beaker in jar test apparatus. Then, the stirrer speed was set at a fast rate (300 rpm/min) and a slow rate (150 rpm, 15 min). After sedimentation, the samples were gathered for the analysis from 1 cm below the water surface and were filtered through the Whatman No.1 filter paper [1].

The residual metal ions were measured by inductively coupled plasma mass spectrometry (Thermo iCAP 6000). The eliminated Cr(VI) and Ni ions were assessed utilizing Formula 1 [4].

$$1.\ \text{Removal}\ \%=\frac{\text{Ci-Ce}}{\text{Ci}}\times 100$$

The obtained coefficient of determination (R^2) was higher than 0.999. The values of the Limit of Detection (LOD) of Cr and Ni were 0.0024 µg/ml and 0.0037 µg/ml, respectively, while Limits of Quantification (LOQ) were found to be 0.0073 µg/ml and 0.0113 µg/ml for Cr and Ni, respectively.

3. Results and Discussion

Removal efficiency in contact time

For achieving ideal time, contact time as a significant factor was changed while different factors, like temperature (25 ºC), coagulant dose (0.1 g/L), blending rate (300

Table 1. The initial concentration of metal ions at a temperature of 25±1 °C

Metal Ions	Initial Concentration (ppm)	Metal ions	Initial Concentration (ppm)	Metal Ions	Initial Concentration (ppm)	Metal Ions	Initial Concentration (ppm)
Ag	0.68	Al	0.48	As	<0.01	Pb	0.44
Ba	0.35	Ni	556.4	Bi	<0.01	Cd	<0.01
Fe	46.7	Cr	86.39	Co	<0.01	Cu	2
Hg	0.02	Mo	<0.01	Sn	0.47	Sb	0.18

Table 2. Physical and chemical properties of wastewater at a temperature of 25±1 °C

Parameter	Results
pH	2.6
EC	5312.75 µs/cm
COD	254 mg/L
BOD	163 mg/L
TDS	4251 mg/L
C:N	11
Temp.	25 ºC

rpm for 1 min and 150 rpm for 15 min), and pH 2.6 were kept consistent. Figures 2 and 3 show the Ni removal productivity influenced by the sedimentation times. According to the results, the Ni removal efficiencies at contact times decreased from 30 to 120 (Figure 2). For more accuracy, the removal efficiencies at contact times of 40 min and 50 min were also examined, which demonstrated a significant increase in 40 min and a subsequent decrease in 50 min (Figure 3); afterward, a contact time of 40 min was chosen as the ideal time with the highest Ni ions removal percentage. In a comparative condition, the removal efficiencies of Cr in sedimentation time had a reduction from 30 to 120 min (Figure 4). Therefore, the removal efficiencies of Cr at 40 and 50 min were additionally examined. Due to the increase and decrease of Cr removal percentage at 40 min and 50 min, respectively, the ideal time was found to be 40 min (Figure 5). Kumar et al. [19] in their research considered 30-45 min as the suitable sedimentation time in wastewater treatment by ferrous sulfate. Qin et al. announced that the most extreme removal efficiencies of Cr and Ni ions by iron coagulants were seen at 40 min [21].

Removal efficiency at different pH values

The chemistry of the coagulant is influenced during coagulation by the pH of the solution. When the coagulants are added to wastewater, the diverse hydrolysis items, including monomers, oligomers, and polymeric hydroxyl buildings are shaped at various pH values [22]. Subsequently, the pH of the solution is one of the considerable parameters in wastewater treatment by coagulants. Low or high pH values can influence treatment proficiency; then, deciding the ideal pH values is fundamental [23]. The experiments for ideal pH were completed at 100 ml of the plating wastewater at various estimations of 1.5, 3, 5, 7, 9, and 11, at a temperature of 25 °C, blended speed of 300 rpm for 1 min and 150 rpm for 15 min, settlement time of 40 minutes, and coagulant dose of 0.1g. Because the study was performed on real wastewater, the initial pH was entered in the diagrams.

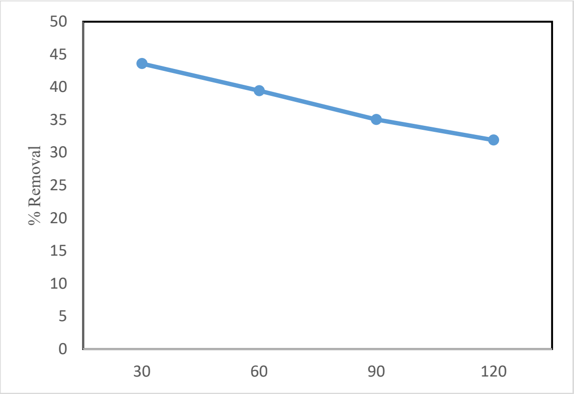


Figure 2. The removal efficiency of Ni(II) by ferrous sulfate at different contact times (30,60,90, and 120 min)

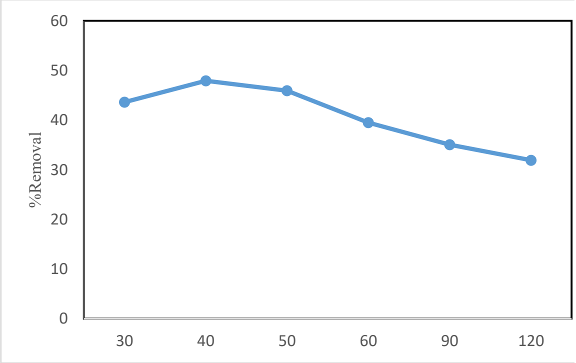


Figure 3. The removal efficiency of Ni (II) by ferrous sulfate at different contact times (30, 40, 50, 60, 90, and 120 min)

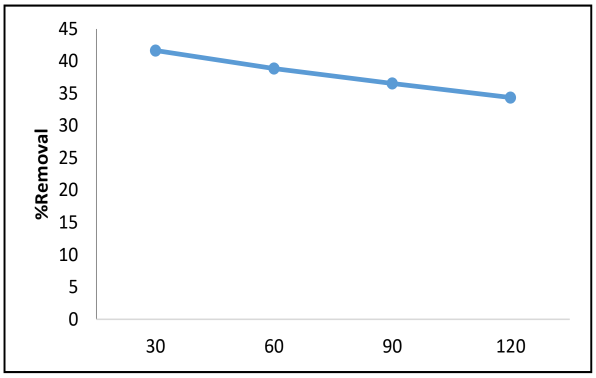


Figure 4. The removal efficiency of Cr (VI) by ferrous sulfate at different contact times (30, 60, 90, and 120 min)

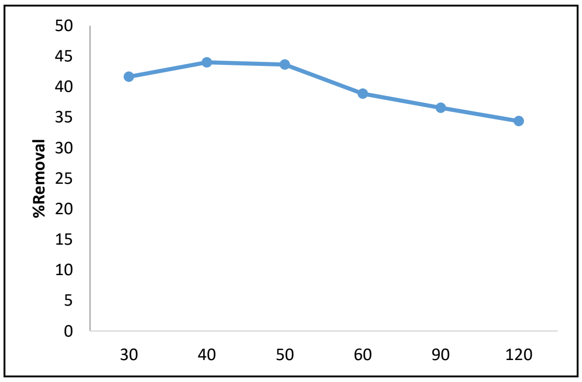


Figure 5. The removal efficiency of Cr (VI) by ferrous sulfate at different contact times (30, 40,50,60,90,and 120 min)

Figures 6 and 7 show the results of these tests. The ideal removal was acquired at pH 7 for Cr(VI) ions (Figure 6). Ferrous sulfate can catch Cr(VI) ions in neutral conditions more than acidic or soluble conditions. In a comparable report, Angadi et al. [24] announced that the most extreme removal efficiencies of Cr ions by alum were seen at pH=7. In another investigation, Prakash et al. [25] detailed that pH=7 is ideal for the removal of turbidity by ferrous sulfate. Likewise, as indicated by Guan et al. [26], the pH=7 can result in the most extreme removal of Cr ions by Fe(II). Katsoyiannis et al. revealed that pH=7 was ideal for the removal of Cr(VI) by ferrous sulfate [27].

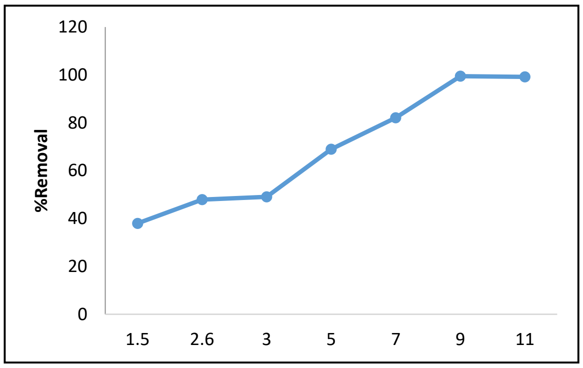


Figure 6. The removal efficiency of Cr (VI) by ferrous sulfate at different pH values

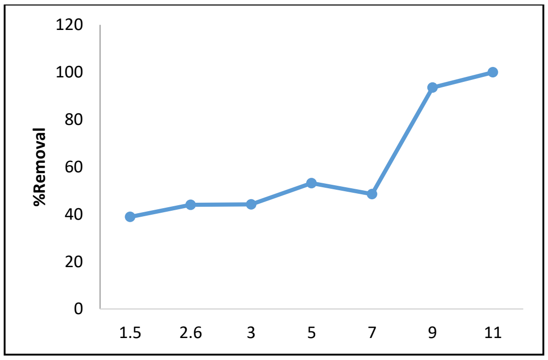


Figure 7. The removal efficiency Ni (II) by ferrous sulfate at different pH values

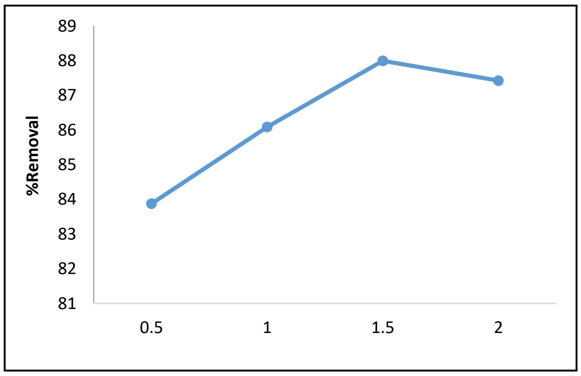


Figure 8. The removal efficiency Cr (VI) by ferrous sulfate at different ferrous sulfate dosages (g/L)

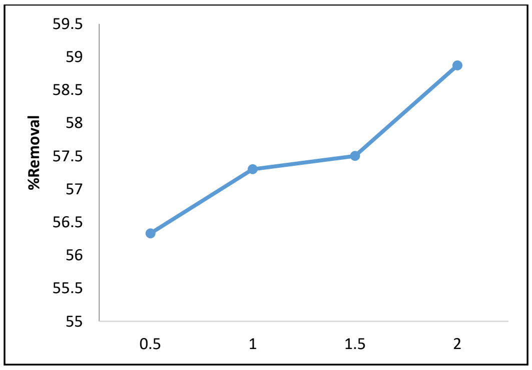
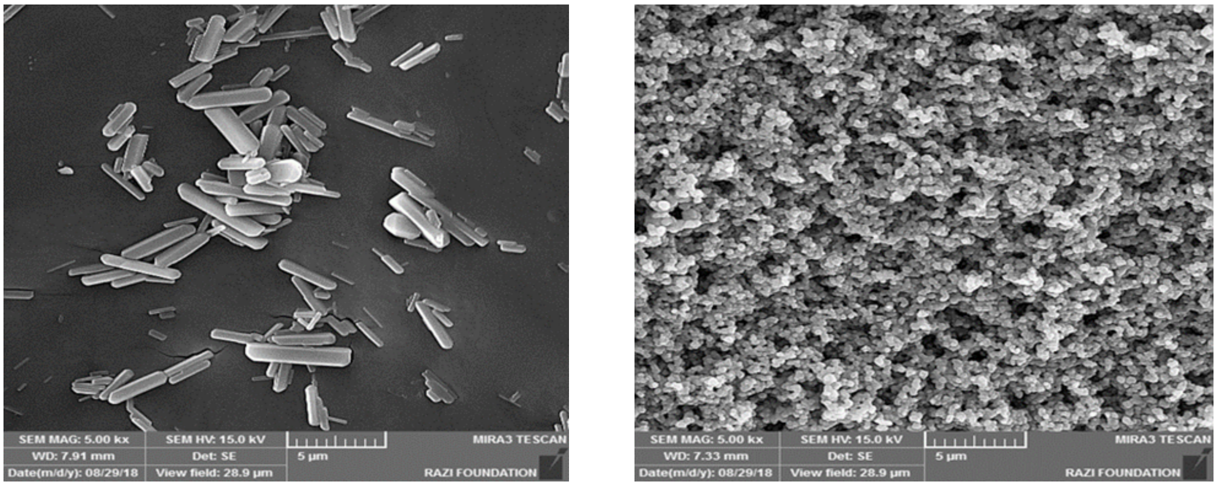


Figure 9. The removal efficiency Ni (II) by ferrous sulfate at different ferrous sulfate dosages (g/L)



a b

Figure 10. SEM micrographs of the ferrous sulfate before (a) and after (b) coagulation

For Ni ions, the ideal removal was reported at a pH of 5. Figure 7 indicates that ferrous sulfate can capture Ni ions in acidic conditions more than basic conditions. Comparative results were reported by Dermentzis et al. [10] for electrocoagulants. In another study, Vlachou et al. [28] reported that the best results for Ni ions removal were observed at an initial pH of 5 by Fe electrocoagulation. Irfan et al. indicated that the ideal pH for COD removal by alum and ferrous sulfate was 5 [29].

The arrangement pH influences the compound coagulation-flocculation process by balancing (1) H+ and metal hydrolysis products for association with organic ligands; and (2) hydroxide particles and organic anions for communication with metal hydrolysis products [3].

As effectively expressed, because heavy metals precipitate as insoluble hydroxides at a pH over 8 [4] and metal particles hydrolyze and precipitate at higher pH esteems from 7-8 [30], a pH of more than 7 was not considered as an ideal pH with high Cr and Ni ions removal percentage.

Removal efficiency at distinct coagulant doses

Changes in Ni ions removal by differing the coagulant measurements (0.5 to 2 g) were observed at 40 min and pH of 5, and for Cr ions, at pH=7 and at 40 min. During the addition of ferrous sulfate to the samples, a higher level of the removal of Cr and Ni ions was observed. The impacts of coagulant dosage on the removal of Cr and Ni ions are demonstrated in Figures 8 and 9. The results confirmed that the removal of Ni and Cr ions is increased for the two particles upon the increase in coagulant dosage.

The results for Cr(VI) ions demonstrated that augmenting the coagulant measurements from 0.5 to 1.5 g/L is associated with the gentle expansion in the removal profi-

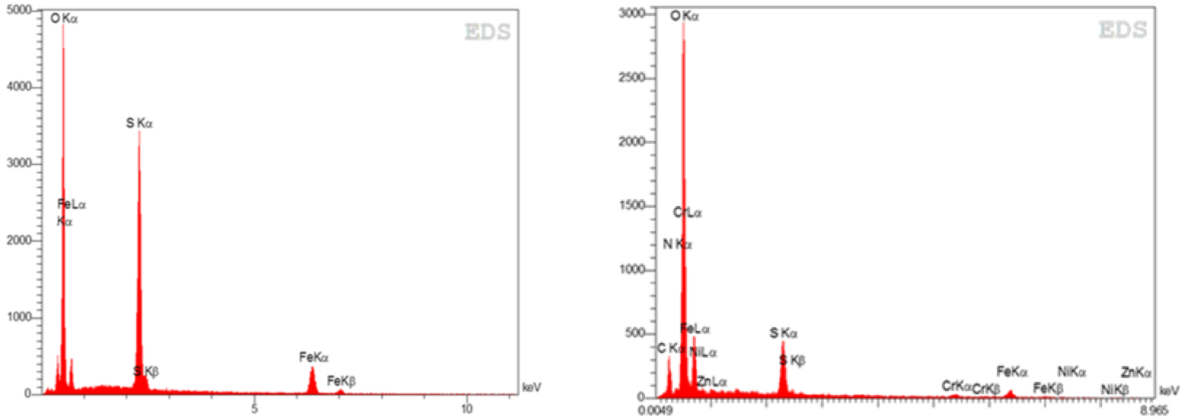


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Figure 11. EDX of the ferrous sulfate before (a) and after (b) coagulation

ciency of Cr ions from 83.88% to 88% (Figure 8). It was seen that there is a slight decline from 1.5 to 2 g/L. The difference between the efficiencies of 0.5 to 1.5 g/L ferrous sulfate was about 4%; therefore, the dose of 0.5 g was considered because of the optimal economic aspect.

Additionally, the results for Ni ions demonstrated that at the coagulant dose of 0.5 to 2 g/L, the removal efficiency is increased from 56.33% to 58.87% (Figure 9). Because the difference between the efficiencies of these two concentrations was about 2%; therefore, 0.5 g/L of ferrous sulfate was determined as the ideal economic dosage for the removal of Ni ions. Although the level of the removal of the metals in the raw water sample increases by adding mg/L of coagulant, for the two metals, the increase process would be delayed after the dosage of 0.5 g/L. Amuda et al. [17] indicated that the optimum dosage for the removal of Cr, Fe, and Zn from leachate by ferric chloride was 0.5 g/L. Also, Bui et al. [23] indicated that the optimum dosage for color, turbidity, COD, and total phosphorus removal from slaughterhouse wastewater by PAC was 0.550 g/L. In another study, Wang et al. [31] obtained the best results for COD and Total Suspended Solids (TSS) removal at 0.5 g/L by ferrous sulfate and alum.

SEM results

The Scanning Electron Microscopy (SEM) results were utilized to break down the superficial structure of ferrous sulfate while coagulation (Figure 10). The analysis uncovered significant data on surface morphology. The SEM images were taken with magnifications of 50 x, 500 x, 1 kx, 5 kx, and 10 kx. They obviously uncovered the surface and morphology of the ferrous sulfate with the magnification of 5 kx. In these images, structures with large superficial regions were apparent. The results showed the contrast between the coagulant surface before and after loading of ions and the coagulation. The coagulation was framed in an adherent crystal shape. Wołowiec et al. obtained similar results to remove heavy metals by Fe salts. The SEM results revealed a typical microcrystalline-organogenic microstructure with a crystal shape [32].

The EDX investigation of ferrous sulfate during coagulation showed that the components present on the surface of the ferrous sulfate before coagulation were N, Fe, S, and O (Figure 11). However, the components on the surface of ferrous sulfate after coagulation were Cr, Ni, C, O, Fe, S, N, and Zn. These changes showed the removal of heavy metals.

4. Conclusions

According to the results, the removal efficiency declined at the contact time of shorter and longer than 40 min. Therefore, a contact time of 40 min can be the ideal time for the removal of Cr(VI) and Ni (II) ions. Additionally, the optimum removal happened at a pH of 7 and 5 for Cr(VI) and Ni(II) ions. For economic reasons, 0.5 g/L of ferrous sulfate for the removal of Cr(VI) and Ni(II) ions would be better. The SEM and EDX results demonstrated the effectiveness of ferrous sulfate in removing Cr(VI) and Ni(II) ions from metal plating wastewater. Thus, ferrous sulfate can be considered an environmentally friendly, cost-effective, easy to use, and effective coagulant with low sludge. It is suggested as a reasonable choice for plating industrial wastewater treatment, especially for Cr(VI) removal with a normal intensity of 83.88% than Ni(II) removal with an intensity of 56.33%. It is recommended that the experiments be performed using a synthetic sample and the results be compared.

Ethical Considerations

Compliance with ethical guidelines

This study was approved by the ethics committee of Islamic Azad University, Hamedan Branch (Code: 17150508951004).

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Authors' contributions

All authors equally contributed to preparing this article.

Conflict of interest

The authors declared no conflict of interest.

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