

The Effect of KMnO_4 , EDTA and CN^- on the Removal of Heavy Metals from Chemical Laboratory Wastewater by Ferritisation

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Abstract

The efficiency of the "ferrite process" for decontamination of copper, nickel and zinc from synthetic wastewater and chemical laboratory wastewater was evaluated. The effect of KMnO_4 , EDTA and CN^- on the decontamination was observed. The initial concentration of heavy metal in synthetic wastewater was 200 mg l^{-1} . The iron amount used for each treatment was $1,400 \text{ mg l}^{-1}$. The experimental conditions were O_2 flow rate of 100 ml min^{-1} , pH 10, temperature of 50°C and treatment time of 45 min. The percentage of removed Cu, Ni and Zn from synthetic wastewater containing those three heavy metals was $> 99.7 \%$. Ethylene diamine tetraacetic acid affected the removal of Ni and significantly, but affected the percentage of removed Cu and Zn slightly. A black sludge of metal ferrite was obtained when having EDTA in low concentration ($\leq 1 \times 10^{-4} \text{ mole l}^{-1}$). A red brown colloid was formed when EDTA was $\geq 5 \times 10^{-4} \text{ mole l}^{-1}$. The higher the EDTA concentration the higher the amount of this red brown colloid. Potassium permanganate caused no effect on the percentage of removed heavy metal. The sludge caused by treatment of wastewater with KMnO_4 in range of $4 \times 10^{-5} - 4 \times 10^{-3} \text{ mole l}^{-1}$ was a black sludge of metal ferrite with magnetic property and quick precipitation, but the sludge caused by treatment of wastewater with $\text{KMnO}_4 \geq 4 \times 10^{-2} \text{ mole l}^{-1}$ was a red violet sludge with no magnetic property. The concentration of CN^- in wastewater was low ($\leq 10 \text{ mg l}^{-1}$), so heavy metal in wastewater could form a complex with CN^- slightly. The percentage of removed Cu, Ni and Zn from chemical laboratory wastewater was $88.92 \pm 0.07 \%$, $87.29 \pm 0.06 \%$ and $98.68 \pm 0.03 \%$, respectively.

Keywords: copper, nickel, zinc, iron, precipitation, recycle, decontamination, hazardous waste

1. Introduction

The amount of wastewater from chemical laboratories increases every year due to the increasing number of students. The wastewater contains various heavy metals in high concentration. In addition it contains KMnO_4 and EDTA in significant amounts and others such as CN^- in low concentration. Technologies available for treating metal contaminated wastewater include chemical oxidation and reduction, precipitation as hydroxides, sulphides and xanthates, chemical displacement, evaporation, ion exchange, membrane separation and adsorption [1-6]. Precipitation, chemical oxidation and reduction, chemical

displacement and adsorption processes generate other forms of hazardous wastes which also require highly regulated and costly disposal. Ion exchange, evaporation and membrane separation processes do not present a sludge disposal problem but do not appear to be economical. Several attempts have been made to precipitate the heavy metals as ferrites [7-15]. This process results in the formation of a ferrite sludge with magnetic properties. Its strong magnetic properties leads to a variety of applications, such as their use as toners for laser printing, pigments, magnetic bands, oil recovery from residues [16].

The aim of the study was to determine the effect of KMnO_4 , EDTA and CN the forming of metal ferrite. The experiments were done on synthetic wastewater containing Cu, Ni and Zn. The results were compared with ferritisation of chemical laboratory wastewater.

2. Materials and Methods

All glassware were washed with detergent in an ultrasonic bath for 1 hour and dried at 100°C for 30 minutes followed by 2 hours at 220°C in an oven. All reagents were of analytical grade and were used without further purification. EDTA, KMnO_4 , $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $\text{FeSO}_4 \cdot 5\text{H}_2\text{O}$ were purchased from Carlo Erba. NaOH, HNO_3 and $\text{ZnSO}_4 \cdot \text{H}_2\text{O}$ were purchased from BDH Laboratory Supplies. KCN, H_2SO_4 and $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ were purchased from Merck. Stock Cu(II), Ni(II) and Zn(II) solution ($1,000 \text{ mg l}^{-1}$) were prepared prior to synthetic wastewater preparation and kept under acid conditions by adjusting the pH to 2 using H_2SO_4 . Four types of synthetic wastewater were prepared. The first type was a wastewater containing only Cu, Ni and Zn. The mixed heavy metal concentration was 200 mg l^{-1} (66.67 mg l^{-1} each). The second type was a wastewater containing Cu (66.67 mg l^{-1}), Ni (66.67 mg l^{-1}), Zn (66.67 mg l^{-1}) and EDTA (concentration varied from $1 \times 10^{-5} - 1 \times 10^{-2} \text{ mol l}^{-1}$). The third type was a wastewater containing Cu (66.67 mg l^{-1}), Ni (66.67 mg l^{-1}), Zn (66.67 mg l^{-1}) and KMnO_4 (concentration varied from $4 \times 10^{-5} - 2 \times 10^{-1} \text{ mol l}^{-1}$). The last type was a wastewater containing Cu (66.67 mg l^{-1}), Ni (66.67 mg l^{-1}), Zn (66.67 mg l^{-1}) and KCN (CN⁻ concentration varied from $0.5 - 10 \text{ mg l}^{-1}$). The chemical laboratory wastewater used was from the chemical laboratory of Chemistry division, Julaporn building, Faculty of Science, King Mongkut's Institute of Technology Ladkrabang. Deionized water was used for preparation of all standards, synthetic wastewaters and reagent solutions.

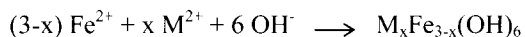
The experimental setup used in the present study is schematically given by Figure 1. The reactor consisted of a 1 liter pyrex beaker, mechanical stirrer, water bath with thermostat, pH meter (Denver Instrument model 215) and oxygen supplying unit. The 500 ml wastewater was placed into the reaction beaker. Subsequently, 3.03 g of $\text{FeSO}_4 \cdot 5\text{H}_2\text{O}$ was added into the solution. The reaction temperature was

controlled to 50°C using a water bath with thermostat. The pH of the solution was continuously measured and adjusted to 10 ± 0.04 using sodium hydroxide. The 99.97 % oxygen was purchased from Thai Industrial Gas Trading Company and continuously passed through the reactor for oxidation. The flow rate was controlled to 100 ml min^{-1} . The content of the reactor was continuous and uniformly mixed by means of a mechanical stirrer. The reaction time was 45 min. After treatment, the color of the obtained sludge was observed. The liquid was separated from the solid by vacuum filtration and GF/C filter paper. The pH of an aliquot was adjusted to below 2 using nitric acid. The heavy metal concentration in an aliquot was quantified by Atomic Absorption Spectroscopy (Shimadzu model AA-680). The obtained solid was analyzed by scanning electron microscopy (Leo model 1455 VP). Three replications for all experiments were performed to test the repeatability and standard deviation.

3. Results and Discussion

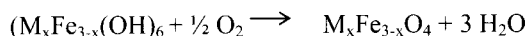
3.1 Experiments performed on synthetic wastewater containing Cu, Ni and Zn

Initial concentration of heavy metal in synthetic wastewater was 200 mg l^{-1} . Given conditions used in this experiment were Fe^{2+} concentration of $1,400 \text{ mg l}^{-1}$, O_2 flow rate of 100 ml min^{-1} , pH 10, temperature of 50°C and contact time of 45 min. The percentages of removed Cu, Ni, Zn and Fe were $99.85 \pm 0.20 \%$, $99.87 \pm 0.17 \%$, $99.72 \pm 0.03 \%$ and $100 \pm 0.00 \%$, respectively. The color of sludge obtained was black. The sludge was tested for its magnetic property by dipping the magnetic bar into wastewater containing that sludge and it that stuck to the magnetic bar. The sludge was then analyzed by scanning electron microscopy. It could affirm that it had the mixing of three metal cations in a metal ferrite structure. In the ferrite process, heavy metal ions in aqueous solution are first coprecipitated with ferrous iron added at a suitable alkaline pH. The forming equation is shown in: [17]:

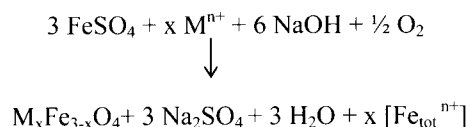


Then, the formed hydroxide mixture is oxidized with oxygen at a temperature $> 40^\circ\text{C}$ until a metal ferrite-bearing precipitate having a

black color and magnetic properties forms. Its reaction is:



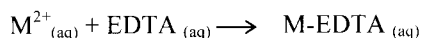
Barrado et al. [18] have suggested the following mechanism for metal ions having divalent and polyvalent metal ions.



3.2 The effect of EDTA

When adding EDTA into synthetic wastewater containing the mixed of three heavy metals, the color of the wastewater would become light blue. And when EDTA amount increased, the color of the wastewater (light blue) would become darker because EDTA reacts with some heavy metals in wastewater. It caused the color of Cu to become obvious. EDTA is tetrapolyprotic acid; $K_1 = 1.02 \times 10^{-2}$, $K_2 = 2.14 \times 10^{-3}$, $K_3 = 6.92 \times 10^{-7}$, and $K_4 = 5.50 \times 10^{-11}$. These K values show that the first two protons come off easier than the last two protons. In an acid solution, EDTA has structure of a neutral ion.

Lone pair electrons in hydroxyl groups and nitro groups cause EDTA molecules to become hexadentate ligands that can assemble with metal ions. Thus, EDTA react with metal ions, and forms a metal – EDTA complex compound as the equation below.



During the treatment of heavy metals in synthetic wastewater containing heavy metals and EDTA, The initial concentration of heavy metal in synthetic wastewater was 200 mg l⁻¹ and the Fe²⁺ concentration used for the treatment was 1,400 mg l⁻¹. Given conditions were O₂ flow rate of 100 ml.min⁻¹, pH 10, temperature of 50°C and treatment time of 45 min. The experimental results was shown in Figure 2.

From the figure it can be seen that when the EDTA concentration increased, the ability to remove metals decreased. EDTA affected the removal of Ni and Fe significantly, but affected the percentage removal of Cu and Zn slightly. When considering industrial wastewater

standard values (< 0.2 mg l⁻¹ for Ni, < 1 mg l⁻¹ for Cu and < 5 mg l⁻¹ for Zn) it is found that the percentage of removed Ni, Cu and Zn were controlled to higher than 99.8 %, 98.5 % and 92.5 %, respectively.

The experiment results found that the percentage of removed Ni was < 99.8 % when the EDTA in wastewater were > 1 x 10⁻⁵ mole l⁻¹; the percentage of removed Cu was < 98.5 % when the EDTA in wastewater was > 1 x 10⁻³ mole l⁻¹; and the percentage of removed Zn was > 97 % when the EDTA was in the range of 1 x 10⁻⁵ - 1 x 10⁻² mole l⁻¹. When considering the characteristics and color of sludge, it was seen that when EDTA ≥ 5 x 10⁻⁴ mole l⁻¹, it was found to be a brown colloid and this red brown colloid would increased when the EDTA was in a high concentration.

Whether there is good formation of metal-EDTA complex or not, this depends on K_f and also the solution pH. In this experiment, synthetic wastewater had a pH of about 3. When adding EDTA and adjusting the pH to lower than 2, EDTA can form complexes with Ni easily. Most metal-EDTA complex compounds are highly stable complex compounds because the forming characteristic of ligand chemical bonds is directional, so it causes metal ions to be in the middle and be surrounded by ligands. Thus, it causes Ni not to be able to form ferrite sludge and be remain in wastewater. The percentage of removed Ni decreases steadily when the EDTA concentration increases.

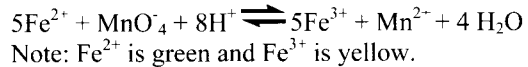
When EDTA concentration increases, the percentages of removed Cu and Zn are likely to decrease slightly because at a range of pH of 2-4, EDTA can form complexes with Ni more, so it causes Cu and Zn to be able to form ferrite sludge normally.

When adding Fe²⁺ and adjusting the pH to 10 ± 0.4, it is seen that Fe²⁺ has an opportunity to form complex with EDTA more than other metals. The remaining iron reacts with heavy metal and form ferrite sludge because irons forms ferrite sludge with other heavy metals that do not form complexes with EDTA. Heavy metals in synthetic wastewater that do not form complexes with EDTA remain in lower concentrations (particular Ni). When the EDTA concentration in wastewater increases, Ni it affects the demand for Fe²⁺ for formation of metal ferrite, so the remaining Fe²⁺ increases.

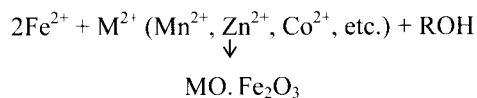
The remaining iron forms a red sludge of iron hydroxide. From the experimental results it is found that there is a black sludge, but also a red one in a kind of colloid. The colloid amount increases when the EDTA concentration in wastewater increases and this sludge is filtered out from wastewater slightly. After filtering out the water it is still red. For this reason the percentage of removed Fe^{2+} decreases when the EDTA concentration increases and when the EDTA is $> 1 \times 10^{-3}$ mole l^{-1} , the percentage of removed iron is $< 96.5\%$ which causes the iron amount in wastewater after treatment to exceed the industrial standard. Barrado et al. [18] studied the effect of metal-binding, organic matter on the efficiency of a students chemistry laboratory wastewater purification with a 5 L reactor and optimized conditions (reaction time, 1 hr; temperature, 60°C ; air flow, 30 l min^{-1} ; pH, 10; $[\text{Fe}^{2+}]/[\text{total metal}]$, 15/L). They found that EDTA concentrations higher than 10^{-3} M decrease purification efficiencies below 99 %. They also found that EDTA concentrations were demonstrated to notably modify the crystalline phase present in ferrite sludge samples. The occurrence of thenardite and goethite increased with increasing EDTA concentration.

3.3 The effect of KMnO_4

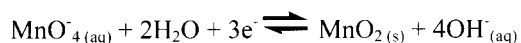
Potassium permanganate is widely used in chemistry and modifies the chemistry of the elements and their oxidation states, thus simulating different levels of redox properties of laboratory effluents. During the treatment of heavy metals in synthetic wastewater with KMnO_4 . The initial concentration of heavy metals in synthetic wastewater was 200 mg l^{-1} and the Fe^{2+} concentration used for the treatment was the 1,400 mg l^{-1} . The given conditions were an O_2 flow rate of 100 ml min^{-1} , pH 9, temperature of 50°C , and contact time of 45 min. When the KMnO_4 concentration in wastewater increased, its color of violet would be darker. After adjusting the pH to < 2 and then adding Fe^{2+} , wastewater containing KMnO_4 (4×10^{-5} mole l^{-1}) it becomes a clear solution, that contains KMnO_4 (4×10^{-5} mole l^{-1}) it becomes a clear green solution. When it contains KMnO_4 (4×10^{-3} mole l^{-1}) it becomes a clear yellow solution. All these are caused because KMnO_4 is a good oxidizing agent and when it is in acid or base condition, KMnO_4 oxidizes Fe^{2+} to Fe^{3+} as the equation below.



The experimental results in Table 1 show that the percentage of removed Cu, Ni, and Zn were $> 99.6\%$ showing that KMnO_4 caused no effect on the removal of heavy metals because the effect of added KMnO_4 is to oxidize Fe^{2+} to Fe^{3+} which increased the speed of oxidation and formation of ferrite sludge [19]. The sludge caused by treatment of wastewater with KMnO_4 in range of $4 \times 10^{-5} - 4 \times 10^{-3}$ mole l^{-1} was a black sludge of metal ferrite with magnetic property and quick precipitation, and the wastewater after treatment was clear. But the sludge caused by treatment of wastewater with $\text{KMnO}_4 > 4 \times 10^{-2}$ mole l^{-1} with a red violet sludge with no magnetic property. This shows that KMnO_4 affected the formation of ferrite sludge. Another problem found after treatment of this wastewater with ferrite was that the Mn^{2+} concentration exceed the specified industrial standard (≤ 5 mg l^{-1}). When the KMnO_4 concentration is low ($4 \times 10^{-5} - 4 \times 10^{-3}$ mole l^{-1}), the Mn^{2+} concentration remains in wastewater slightly. KMnO_4 oxidizes Fe^{2+} to Fe^{3+} and forms Mn^{2+} that can form metals ferrite and also assemble with other metals as the equation below.



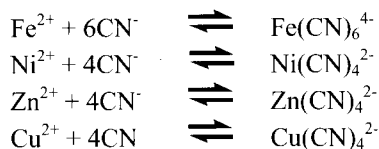
When the KMnO_4 concentration is $> 4 \times 10^{-2}$ mole l^{-1} , it is mainly Mn^{2+} and the obtained sludge is red violet. This is not ferrite sludge. When having a high KMnO_4 amount and the Fe^{2+} amount is limited, The MnO_4^- amount remains high. The remaining MnO_4^- amount may interfere and block the formation of trivalent and divalent hydroxo complexes that affect the forming reaction of ferrite sludge the can not be formed normally. The forming reaction forms only the early step, that is the formation of metal hydroxide sludge, but some KMnO_4 forms MnO_2 as the equation below.



3.4 The effect of CN⁻

During the treatment of heavy metals in synthetic wastewater, with CN⁻ the initial concentration of heavy metals in synthetic wastewater was 200 mg l⁻¹ and the Fe²⁺ concentration used for the treatment was 1,400 mg l⁻¹. The given conditions were O₂ flow rate of 100 ml min⁻¹, pH 9, temperature of 50°C and contact time of 45 min. From the experimental results in Table 2 it can be seen that the sludge from the treatment of wastewater with CN⁻ in a concentration range of 0.5–10 mg l⁻¹ is pitch-black with magnetic property which is a characteristic of ferrite sludge. And the percentage of removed three heavy metals decreased slightly when compared to the treatment of wastewater with CN⁻.

This is caused by complexity with CN⁻ which uses 1 mole of heavy metal 1 mole to react with 4–6 mole of CN⁻. This experiment only has 0.5 to 10 mg l⁻¹ CN⁻, so heavy metals in wastewater form complexes with CN⁻ (as equation below) slightly [20].



The remaining heavy metals can form ferrite sludge normally.

3.5 Experiments performed on chemical laboratory wastewater

Physical and chemical characteristics of chemical laboratory wastewater are shown in Table 3. The given conditions used in this experiment were Fe²⁺ concentration of 1,400 mg l⁻¹, O₂ flow rate of 100 ml min⁻¹, pH 10, temperature of 50°C, and contact time of 45 min. The percentage of removed Cu, Ni, Zn and Fe were 88.92 ± 0.07 %, 87.29 ± 0.06 %, 98.68 ± 0.03 % and 90.72 ± 0.05 %, respectively. Only a little black sludge was obtained. This was mostly found in the form of a red colloid (iron hydroxide). This colloid was not easily precipitated and had no magnetic property. The percentages of removed Cu, Ni, Zn and Fe when treating synthetic wastewater were very much different from when treating chemical laboratory wastewater (Figure 3).

4. Conclusions

The ferrite process can be a good alternative to treat wastewater containing multiple heavy metals with low concentration of EDTA, KMnO₄ and CN⁻. The percentage of removed metal from synthetic wastewater containing only Cu, Ni and Zn was > 99.7%. High concentrations of EDTA and KMnO₄ affected the formation of ferrite sludge. A black sludge of metal ferrite was obtained when the concentration of EDTA was ≤ 1 × 10⁻⁴ mole l⁻¹ and the concentration of KMnO₄ was ≤ 4 × 10⁻³ mole l⁻¹. Ethylene diamine tetraacetic acid affected the removal of Ni and Fe significantly, but affected the percentage removal of Cu and Zn slightly. The percentage of removed Ni was as low as 24.28 % and 0.28 % when EDTA concentrations were 5 × 10⁻³ mole l⁻¹ and 1 × 10⁻² mole l⁻¹, respectively. From this experiment, the lowest percentage of removed Fe was 69.59 %. More than 97% of copper and zinc were removed when having EDTA in concentration between 1 × 10⁻⁵ mole l⁻¹ to 1 × 10⁻² mole l⁻¹. Potassium permanganate caused no effect on the percentage of removed heavy metals. The concentration of cyanide used in this experiment was too low to observe the effect. The percentages of removed Cu, Ni and Zn from chemical laboratory wastewater were 88.92 ± 0.07 %, 87.29 ± 0.06 % and 98.68 ± 0.03 %, respectively.

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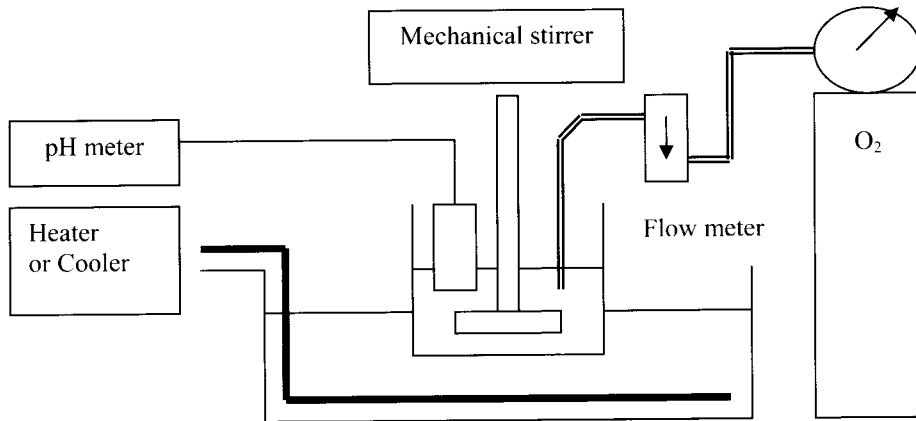


Figure 1 A laboratory scale reactor

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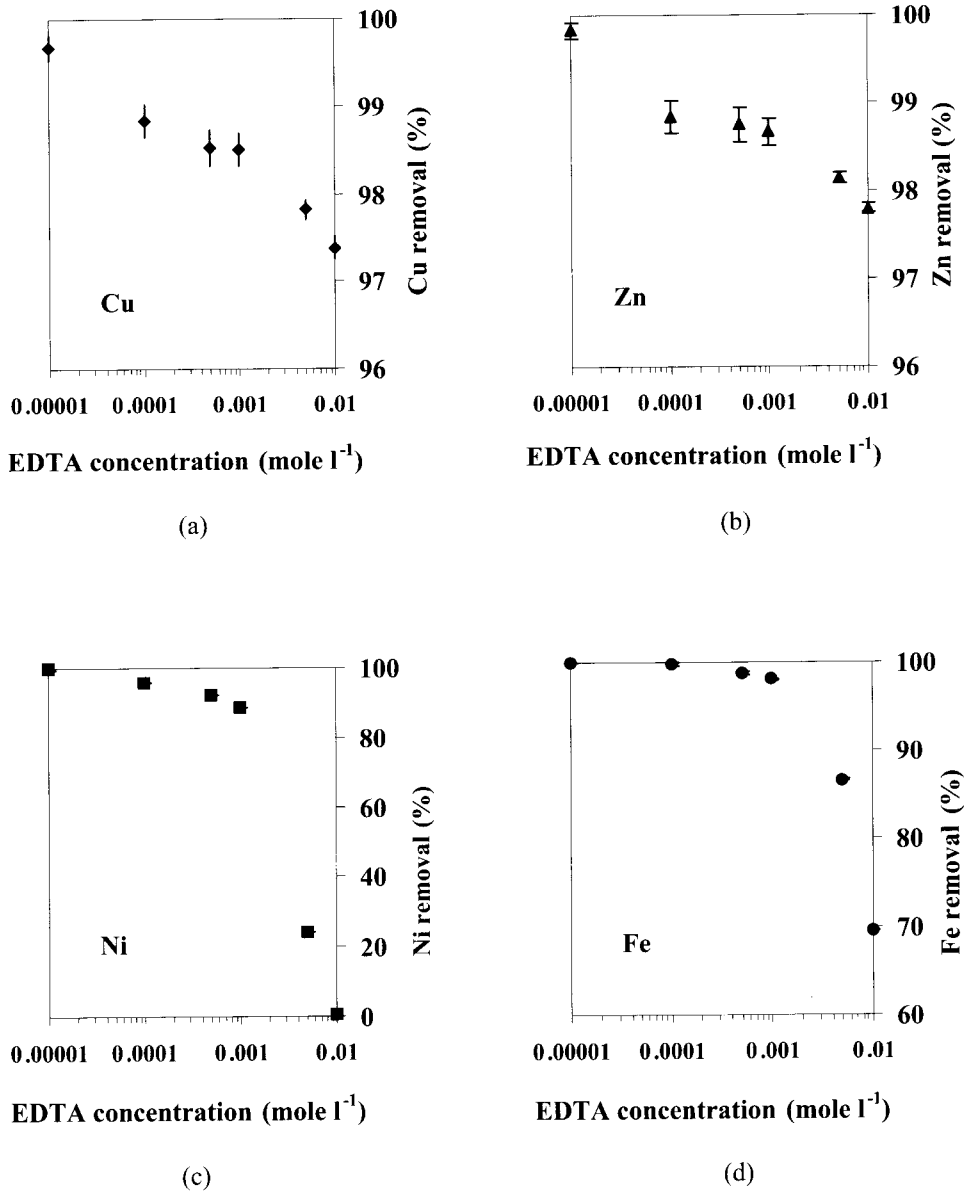


Figure 2 Relationship between EDTA concentration and percentage of removed metal

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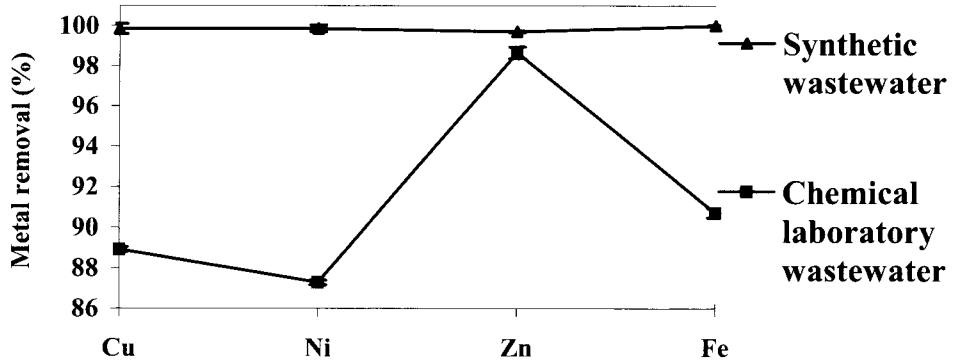


Figure 3 The difference between the removal of metal from synthetic wastewater containing only 3 heavy metals and chemistry laboratory wastewater

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Table 1 The percentage of metal removal, Mn²⁺ concentration and sludge characteristic when varying KMnO₄ concentration

KMnO ₄ conc. (mole l ⁻¹)	[Mn ²⁺] (mg l ⁻¹)		Sludge characteristic	Percentage of metal removal			
	initial	final		Cu ²⁺	Ni ²⁺	Zn ²⁺	Fe ²⁺
4 x 10 ⁻⁵	2.20	0.06	Black sludge, clear solution, with magnetic property	99.06 (± 0.11)	99.80 (± 0.16)	99.72 (± 0.12)	99.91 (± 0.10)
4 x 10 ⁻⁴	22.20	0.35	Black sludge, clear solution, with magnetic property	99.90 (± 0.10)	99.90 (± 0.13)	99.83 (± 0.09)	99.92 (± 0.08)
4 x 10 ⁻³	222.00	0.46	Black sludge, clear solution, with magnetic property	100.00 (± 0.04)	99.92 (± 0.18)	99.77 (± 0.17)	99.95 (± 0.09)
4 x 10 ⁻²	2224.00	1095.24	Red violet sludge, no magnetic property	100.00 (± 0.02)	99.85 (± 0.14)	99.62 (± 0.12)	99.91 (± 0.10)
2 x 10 ⁻¹	10988.00	6455.00	Red violet sludge, no magnetic property	99.91 (± 0.08)	99.74 (± 0.09)	99.66 (± 0.08)	99.92 (± 0.09)

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Table 2 The percentage of metal removal when varying CN^- concentration

CN ⁻ conc. (mg l ⁻¹)	Percentage of metal removal			
	Cu ²⁺	Ni ²⁺	Zn ²⁺	Fe ²⁺
0.5	100.01 ± 0.02	99.95 ± 0.06	99.82 ± 0.11	99.99 ± 0.01
1	99.58 ± 0.07	99.86 ± 0.04	99.99 ± 0.05	100.00 ± 0.01
2	99.30 ± 0.11	99.76 ± 0.10	99.68 ± 0.12	100.00 ± 0.02
5	98.92 ± 0.13	99.74 ± 0.13	98.98 ± 0.14	99.99 ± 0.03
10	98.62 ± 0.05	98.99 ± 0.17	98.79 ± 0.15	99.99 ± 0.01

Table 3 Characteristic of wastewater from chemical laboratory

Parameter	Value
Color	blue green
pH	7.81
Temperature	25°C
EDTA	1 x 10 ⁻³ mol l ⁻¹
KMnO ₄	4 x 10 ⁻³ mol l ⁻¹
Cu	190.04 ppm
Ni	83.16 ppm
Zn	11.22 ppm
Fe	5.27 ppm
Pb	5.27 ppm
CN	< 0.1 ppm