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Fenton and Fenton-like reaction followed by hydroxide precipitation in the removal of Ni(II) from NiEDTA wastewater: A comparative study

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ABSTRACT

It is difficult to remove Ni(II) from NiEDTA wastewater by many conventional processes. A new method for removing Ni(II) from NiEDTA wastewater by Fenton and Fenton-like reaction followed by hydroxide precipitation presents an important breakthrough. This treatment process is referred to as Fenton/Fenton-like reaction-hydroxide precipitation (FR-HP) process. This study investigated the use of FR-HP process for the removal of Ni(II) from NiEDTA complex wastewater. The removal efficiency of Ni(II) by FR-HP process was investigated and the kinetics of the two processes was studied. Results indicated that the removal efficiency was strongly dependent on the initial concentration of Fe^{2+} or Fe^{3+} and H_2O_2, initial and precipitation pH and temperature. Both FR-HP processes can effectively remove Ni(II). At optimal operation conditions, the removal efficiency of Ni(II) were 92.8% and 94.7% for Fenton and Fenton-like systems after 60 min, respectively. FR-HP process seems to be an economically and environmentally friendly process to remove the metal from metal–EDTA wastewater. Hopefully, FR-HP process will promote the development of metal–EDTA treatment technology.

1. Introduction

With the rapid industrialization, the contamination of water resources has been a worldwide environment problem. Nickel, copper, cadmium, zinc, lead, mercury and chromium are often present in industrial wastewaters, being discharged by metal plating, mining activities, smelting, battery manufacture, tanneries, petroleum refining, paint manufacture, etc. [1]. Many heavy metal laden wastewaters contain organic complexing agents, which are widely used in industrial applications for dissolving metals. Ethylenediaminetetraacetate (EDTA) is a widely used complexing agent in plating industry, metal finishing and chemical cleaning processes [2]. EDTA can make it difficult to remove heavy metals in wastewaters by many conventional precipitation processes mainly because of the dramatic increase in the solubility of heavy metal ions. Precipitation technologies for metal removal, such as hydroxide and sulfide precipitation, are inhibited when the metals exist as metal–EDTA complexes [3]. Specialized treatment processes can be employed for metal–EDTA complexes. For example, CuEDTA can be treated by dithiocarbamate-type precipitant [4,5], but in treating electroplating wastewater containing NiEDTA, hydroxide, sulfide and dithiocarbamate-type precipitants all did not work for it. Therefore, searching for a treatment technology which can treat many common metal–EDTA complexes is of interest.

Over the past few decades, advanced oxidation processes (AOPs) have received increasing attention for the effective destruction of a wide range of recalcitrant organic pollutants [6]. Among AOPs, oxidation using Fenton's reagent is an attractive and effective technology for the degradation of a large number of hazardous and organic pollutants because of the lack of toxicity of the reagents, eventually leaving no residues and the simplicity of the technology [7]. The degradation mechanism of organic pollutants by Fenton reaction is shown below [8,9]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {}^{\bullet}OH + OH^- \quad (1)$$

$$RH + {}^{\bullet}OH \rightarrow R^{\bullet} + H_2O \quad (2)$$

$$R^{\bullet} + Fe^{3+} \rightarrow R^+ + Fe^{2+} \quad (3)$$

$$Fe^{2+} + {}^{\bullet}OH \rightarrow Fe^{3+} + OH^- \quad (4)$$

The produced hydroxyl radical is well known as a strong oxidant and can oxidize many recalcitrant organic compounds rapidly. Organic molecules are then degraded into carbon dioxide, inorganic ions and water. Many investigations have been conducted into the degradation of various organics using Fenton oxidation [10,11]. The main drawback of this technique is represented by the cost of the reactants, H_2O_2 and Fe^{2+}. For this reason, various methods have been introduced to use the lower cost Fe^{3+} because Fenton-like oxidation is also effective. The reaction of hydrogen peroxide with

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Fe^{3+} is referred to as a Fenton-like reaction, Eqs. (5)–(8) [12].

$$Fe^{3+} + H_2O_2 \leftrightarrow Fe\cdots OOH^{2+} + H^+ \quad (5)$$

$$Fe\cdots OOH^{2+} \rightarrow HO_2^{\bullet} + Fe^{2+} \quad (6)$$

$$H_2O_2 + Fe^{2+} \rightarrow {}^{\bullet}OH + OH^- + Fe^{3+} \quad (7)$$

$$^{\bullet}OH + RH \rightarrow R^{\bullet} + H_2O \quad (8)$$

However, little work has been reported on the removal of heavy metal ions in metal–EDTA complexes by Fenton and Fenton-like reaction. For this purpose in this study, the potential of a new method for removing heavy metals by Fenton or Fenton-like reaction followed by hydroxide precipitation presents an important breakthrough. This treatment process is referred to as Fenton or Fenton-like reaction-hydroxide precipitation (FR-HP) process in this paper. After metal–EDTA wastewater was treated by FR-HP process, free heavy metal ions were released and precipitated by hydroxide.

This study discusses the conditions for effective oxidation of metal–EDTA complexes and the removal efficiencies of metal ions. For the metal–EDTA complexes, NiEDTA was selected as a target pollutant in consideration of the extensive environmental impact of Ni(II) and that many conventional technologies, such as hydroxide, sulfide and dithiocarbamate-type precipitants can hardly work for it. The removal efficiency of Ni(II) by the two processes was investigated and compared and the kinetics of the two reactions was studied. The reaction parameters affecting the removal efficiency of Ni(II) such as initial concentration of Fe^{2+}, Fe^{3+}, H_2O_2, initial and precipitation pH values and reaction temperature were also investigated.

The objectives of the study were to: (1) investigate the effect of initial parameters on the removal of Ni(II) from NiEDTA by Fe^{2+}/H_2O_2 (Fenton reaction) or Fe^{3+}/H_2O_2 (Fenton-like reaction); (2) reveal the optimum conditions of removal Ni(II) from NiEDTA by FR-HP processes; (3) assess the practical potential for FR-HP processes to perform the metal–EDTA wastewater.

2. Materials and methods

2.1. Materials

The following analytical-grade reagents: 30% hydrogen peroxide (H_2O_2) aqueous solution, $NiSO_4{\cdot}6H_2O$, Na_2EDTA, $FeSO_4{\cdot}7H_2O$, $Fe_2(SO_4)_3$, NaOH and H_2SO_4 were obtained from Tianjin Fuchen Chemical Reagents Factory. The $FeSO_4$ solution was prepared daily. Distilled water was used throughout the research. Initial Ni^{2+} concentrations in NiEDTA solutions were 50.00 mg/l.

2.2. Analytical apparatus and methods

The UV–vis spectra of NiEDTA before and after FR-HP processes were determined using a UV-PC 3101PC spectrophotometer (Shimaszu, Japan). The residual total Ni (Ni^{2+} and NiEDTA) and total Fe (Fe^{3+} and Fe^{2+}) concentrations were analyzed by WFX-210 atomic absorption spectrophotometer (AAS). A PHS-25 pH meter was used to determine pH values of solutions. The configuration of precipitates was analyzed by a Nikon Eclipse E400 Microscope.

2.3. FR-HP processes

All experiments were carried out in 200 ml beakers. Firstly, the beakers were charged with 100 ml NiEDTA solution containing moderate Ni^{2+} concentration of 50.00 mg/l at a temperature of 25–50 °C. Then, 3.0 M H_2O_2 and 0.05 M Fe^{2+} or Fe^{3+} were added into the NiEDTA solution and mixing was continued for 60 min. For most of the experiments, the initial pH was set at 3.0 by a calibrated pH meter. For the experimental runs at different pH, the values were adjusted using sulphuric acid or sodium hydroxide solutions. After the reaction completed, the precipitation experiments were conducted with Jar test method. The Fenton or Fenton-like treated effluents were adjusted to alkaline pH values using 10% of NaOH and were agitated at 80 rpm for 15 min. Then the forming solid precipitates were allowed to settle for 30 min. The effluents were withdrawn and filtered through 0.45 μm Millipore filter. Then the filtrate was analyzed for residual total nickel and total Fe concentrations using AAS. The removal efficiency of Ni(II) by FR-HP processes was calculated by the following relationship:

$$\text{Nickel removal efficiency }(\%) = \frac{C_{Ni,0} - C_{Ni,t}}{C_{Ni,0}} \times 100 \quad (9)$$

where $C_{Ni,0}$ and $C_{Ni,t}$ are the concentrations of total nickel at reaction time 0 and t, respectively.

3. Results and discussion

3.1. EDTA oxidation

Several researchers studied EDTA oxidation and they proposed that the initial oxidation step most likely occurs at the acetate group [3,13]. Fig. 1 presents UV–vis spectral change of NiEDTA in water before and after FR-HP processes. As can be observed, before FR-HP processes, the absorption spectrum was characterized by maximum absorption peak at 208 nm, originating from carboxyl group in NiEDTA. The complete disappearance of the band was due to the fragmentation of EDTA by oxidation, elucidating Fenton and Fenton-like processes were effective to degrade EDTA. In the next experiments, we mainly investigate the total nickel removal.

3.2. Comparison of Fenton and Fenton-like processes

After the Fenton and Fenton-like treated effluents were adjusted to alkaline pH, tan precipitates formed. Fig. 2 shows the photographs of precipitates with 400× magnification. As shown in the figure, there is nearly no difference of the two kinds of precipitates. Ni^{2+}, Fe^{2+} and Fe^{3+} ions are in existence in the Fenton and Fenton-like treated effluents. Under OH^- conditions, the metal ions can form hydroxide precipitate.

Fig. 3 shows a comparison of residual concentration of Ni(II) with two FR-HP processes at 25 °C. From the figure, the removal efficiency of Ni(II) increased fast at first 60 min, and then remained almost unchanged after 60 min. So in the next experiments, reaction time of 60 min was chosen. And it seems that both Fenton and

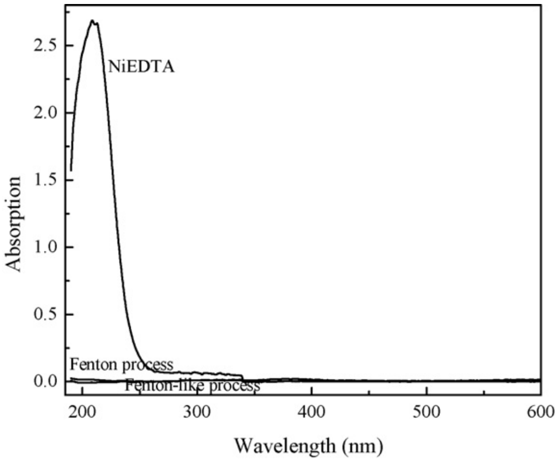

Fig. 1. UV–vis spectral change of NiEDTA solution before and after FR-HP processes.

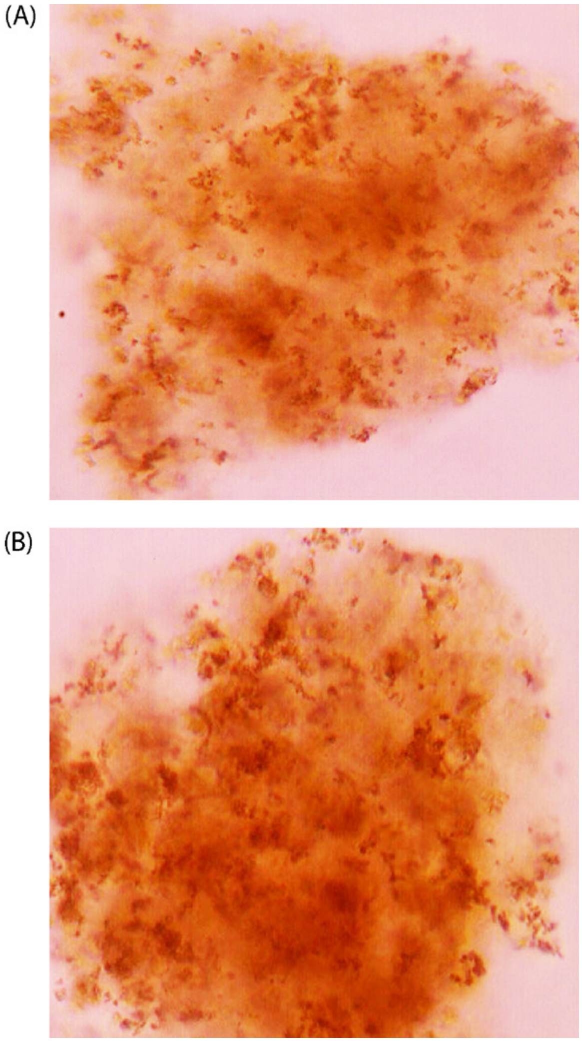

Fig. 2. Microscopic images of three precipitates with 400× magnification: (A) Fenton process; (B) Fenton-like process.

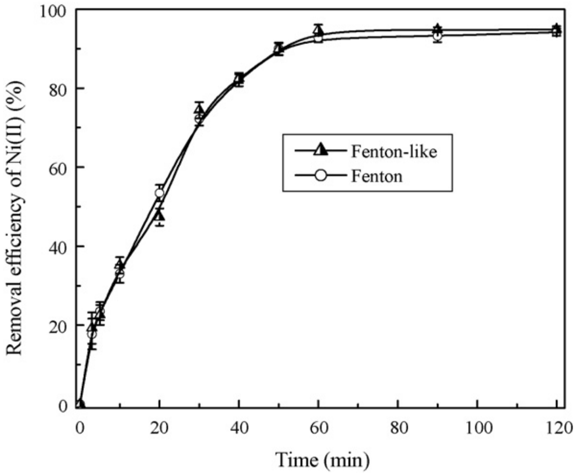

Fig. 3. Comparison of Fenton and Fenton-like processes for the removal of Ni(II) from NiEDTA wastewater containing 50.00 mg/l Ni^{2+}. Reaction conditions: $[Fe^{2+}]_0 = 1$ mM, $[Fe^{3+}]_0 = 1$ mM, $[H_2O_2]_0 = 141$ mM, initial pH 3.0, last pH 11.0 and $T = 25\,^\circ C$. The error bars in the figure indicate the standard deviations which were calculated by replicate data.

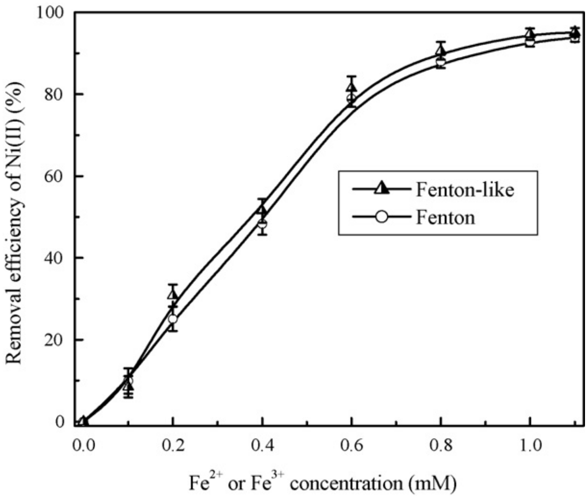

Fig. 4. Effect of initial Fe^{2+} or Fe^{3+} concentration on the removal efficiency of nickel in the treatment of NiEDTA wastewater containing 50.00 mg/l Ni^{2+} by Fenton and Fenton-like reactions. Reaction conditions: $[H_2O_2]_0 = 141$ mM, initial pH 3.0, last pH 11.0 and $T = 25\,^\circ C$. The error bars in the figure indicate the standard deviations which were calculated by replicate data.

Fenton-like processes exhibited similar Ni(II) removal efficiency. For example, at 20, 60 and 120 min, Ni(II) removal efficiency was 48.4%, 92.8% and 94.2% for Fenton system, and 47.3%, 94.7% and 94.8% for Fenton-like system. However, statistical analysis of Ni(II) removal efficiency at different time (except 0 min) by two FR-HP processes in Fig. 3, there are 70% Ni(II) removal efficiency by Fenton-like higher than Fenton system. Therefore, we can conclude that there exists higher Ni(II) removal efficiency for Fenton-like system than Fenton system. In other studies [7,12,14], the degradation rate in Fenton reaction was much faster than that of the Fenton-like reaction in the initial stages. These authors explained that $^\bullet OH$ has stronger oxidation capability than hydroperoxyl radical or the immediate formation of hydroxyl radicals in the Fenton reagent. In our experiments, Fenton-like process presented higher efficiency than Fenton process which can be interpreted by the mechanism of ligand exchange. The extent of exchange related to the metal–EDTA stability constants is listed as follows [15]: $\mathrm{Log}(K)_{\text{Fe(III)-EDTA}} = 27.7$, $\mathrm{Log}(K)_{\text{Fe(II)-EDTA}} = 16.0$, $\mathrm{Log}(K)_{\text{Ni(II)-EDTA}} = 20.1$. Ni^{2+} has an EDTA log stability constant considerably less than Fe^{3+} and is much more strongly bound than Fe^{2+}. Hence, EDTA ligand exchange reaction takes place and frees partly Ni^{2+} from NiEDTA complex. So in Fenton-like system, NiEDTA can release Ni^{2+} through two steps, major oxidation by hydroxy radicals and minor ligand exchange of NiEDTA/Fe^{3+} as shown in following equations:

$$\text{Ni(II)-EDTA} + {}^\bullet\text{OH} \rightarrow \text{Ni}^{2+} + \text{EDTA}_{\text{ox prod}} \tag{10}$$

$$\text{Fe}^{3+} + \text{Ni(II)-EDTA} \leftrightarrow \text{Ni}^{2+} + \text{Fe(III)-EDTA} \tag{11}$$

Therefore, Fenton-like reaction-hydroxide precipitation process presented higher Ni(II) removal efficiency than Fenton process.

3.3. *Effect of Fe^{2+} and Fe^{3+} concentrations*

In order to investigate the effect of Fe^{2+} and Fe^{3+} concentration on the Ni(II) removal efficiency, experiments were conducted at different Fe ions concentrations with 141 mM H_2O_2 solutions. Fig. 4 shows the effect of Fe^{2+} (Fe^{3+}) concentration on nickel removal by Fenton and Fenton-like processes. As can be seen, an obvious increase of removal efficiency was observed by increasing initial Fe^{2+} or Fe^{3+} concentration from 0 to 1.0 mM. When the Fe^{2+} or Fe^{3+} concentration was 1.0 mM, the extent of nickel removal was 92.8% and 94.7% for Fenton and Fenton-like sys-

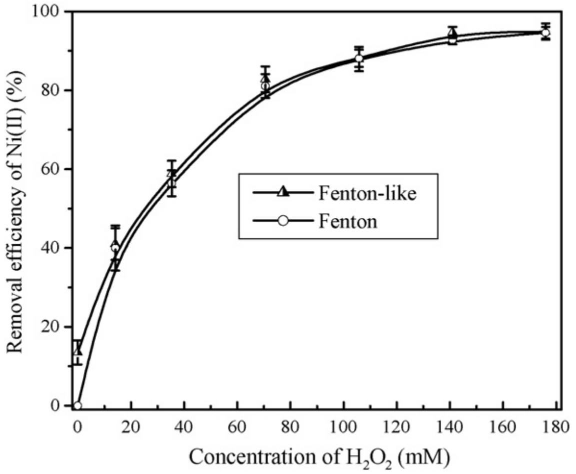

Fig. 5. Effect of initial H_2O_2 concentration on the removal efficiency of nickel in the treatment of NiEDTA wastewater containing 50.00 mg/l Ni^{2+} by Fenton and Fenton-like reactions. Reaction conditions: $[Fe^{2+}]_0 = 1.0$ mM, $[Fe^{3+}]_0 = 1.0$ mM, pH 3.0, last pH 11.0 and $T = 25\,^{\circ}C$. The error bars in the figure indicate the standard deviations which were calculated by replicate data.

tems after 60 min, respectively. However, further increasing Fe^{2+} or Fe^{3+} concentration above 1 mM did not bring about further improvement in the nickel removal. Many studies reported in literature have revealed that the use of a much higher concentration of Fe^{2+} could lead to the selfscavenging of $^{\bullet}OH$ radical by Fe^{2+} [16,17] and induce the decrease in degradation rate of pollutants: $^{\bullet}OH + Fe^{2+} \rightarrow Fe^{3+} + OH^{-}$. Statistical analysis of the data in Fig. 4 also shows that there exists 83.3% Ni(II) removal efficiency by Fenton-like system higher than Fenton under different initial Fe concentration.

3.4. Effect of H_2O_2 concentration

The amount of H_2O_2 is considered one of the most important factors in Fenton and Fenton-like oxidation. The effect of H_2O_2 dose on the removal efficiency of Ni(II) in FR-HP processes was shown in Fig. 5. As can be seen, with no H_2O_2, the removal efficiency of Ni(II) was zero after 60 min in Fenton system, while the removal efficiency of Ni(II) was 12.5% in Fenton-like system. This result can also confirm that there is a ligand exchange reaction in the Fenton-like system. The figure also indicated that with the increase of the amount of H_2O_2, the removal efficiency of Ni(II) increased for the two systems. The increase in the removal efficiency is due to the increase in hydroxyl radical concentration by addition of H_2O_2. When the H_2O_2 concentration increased to 141 mM, 92.8% and 94.7% nickel removal could be achieved after 60 min in the Fenton and Fenton-like systems, respectively. Further increase H_2O_2 amount from 141 to 176 mM causes little increase in Ni(II) removal efficiency. It is important to mention that, the Fenton and Fenton-like systems showed a high H_2O_2 demand, as previously reported by Sundstrom et al. [18] and Ghiselli et al. [6]. De Laat and Gallard [19] have reported that when $C_{H_2O_2}/C_{Fe^{2+}}$ molar ratio is greater than 500, a detrimental effect might be observed. And in this study, the lower ratios (<200) have been employed and detrimental effect has not been observed. Fig. 5 also shows that there exists higher Ni(II) removal efficiency for Fenton-like system than Fenton system under different initial H_2O_2 concentration.

3.5. Effect of pH

Since the pH value is one of the most important control variables in Fenton and Fenton-like processes, its effect on the Ni(II) removal was also investigated at different pH values. From Fig. 6(A), it is evident that when the initial pH increases from 2 to 3, the removal efficiency quickly increases, decreasing when the pH is raised from 3 to 5. The results from this experiment are in agreement with those studies reported by the researchers [8,11,20], who found that acidic pH levels near 3 are usually optimum for Fenton and Fenton-like oxidation. Therefore, for all further experiments a pH of 3.0 was chosen.

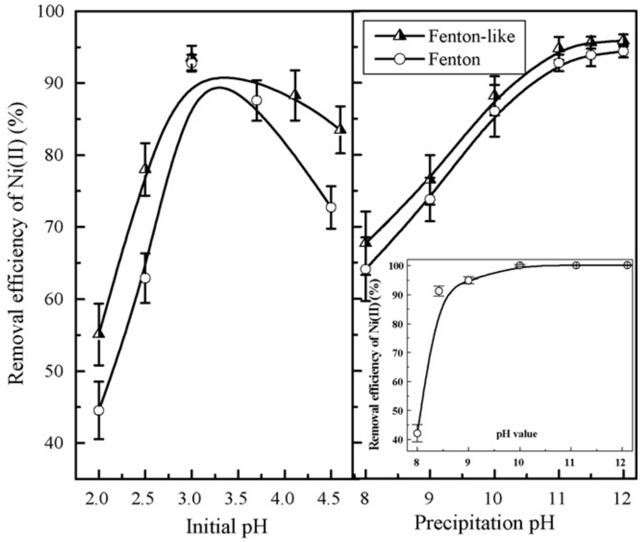

Fig. 6. Effect of initial pH value (A) and precipitation pH value (B) on removal efficiency of Ni(II) in the treatment of NiEDTA wastewater containing 50.0 mg/l Ni^{2+} by Fenton and Fenton-like reactions. Reaction conditions: $[Fe^{2+}]_0 = 1.0$ mM, $[Fe^{3+}]_0 = 1.0$ mM, $[H_2O_2]_0 = 141$ mM and $T = 25\,^{\circ}C$. Inset: pH-solubility equilibrium diagram of $Ni(OH)_2$. The error bars in the figure indicate the standard deviations which were calculated by replicate data.

The lower efficiency below pH 3.0 in Fenton system is due to the formation of $[Fe(II)(H_2O)_6]^{2+}$ complex, which reacts more slowly with H_2O_2 than $Fe(II)(OH)(H_2O)_5]^{+}$, and therefore produces fewer $^{\bullet}OH$ [21]. While in Fenton-like system, at a low pH, the reaction of Fe^{3+} with H_2O_2 is inhibited [22], and $^{\bullet}OH$ could be consumed by the scavenging effects of H^{+} which will limit the degradation rate [23]. And the Ni(II) removal efficiency clearly declined at pH > 3, probably because of the decrease in free iron species in the solution. Actually, at higher pH values iron ion precipitates, therefore the concentration of dissolved Fe^{2+} or Fe^{3+} is decreasing [24]. Also, if the pH is too high, the iron precipitates as $Fe(OH)_3$ and H_2O_2 is decomposed the to oxygen, which reduces its concentration in the solution [25,26].

After NiEDTA wastewater was treated by Fenton or Fenton-like reaction, the solution was adjusted to alkaline pH values to precipitate Ni(II) and Fe ions. As seen in Fig. 6(B), with pH values increasing from 8.0 to 11.0, the Ni(II) removal efficiency increased from 64.1% to 92.8% for Fenton and from 67.7% to 94.8% for Fenton-like system because Ni(II) formed $Ni(OH)_2$ precipitate at higher pH values. When pH increased further, the removal efficiency remained almost unchanged. So in our experiments the adjusted pH 11.0 after Fenton or Fenton-like systems was chosen. And the pH-solubility equilibrium diagram of $Ni(OH)_2$ has also been given in Fig. 6B (inset). From the inset, we can see that the removal efficiency of Ni(II) is higher in OH^{-} than FR-HP systems when $pH \geq 9.0$. This is happens because that Ni^{2+} can compete with Fe^{2+}/Fe^{3+} for OH^{-} to form hydroxide precipitates in FR-HP systems, while in pure OH^{-} condition, only Ni^{2+} reacts with OH^{-} to form $Ni(OH)_2$.

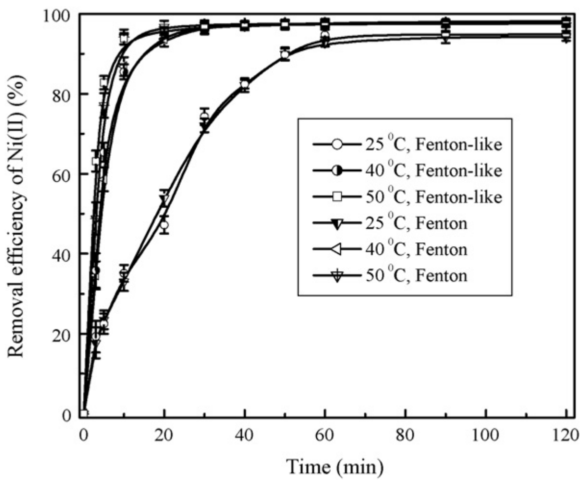

Fig. 7. Effect of temperature on the removal efficiency of nickel ion in the treatment of NiEDTA wastewater containing 50.00 mg/l Ni^{2+} by Fenton and Fenton-like. Reaction conditions: $[H_2O_2]_0$ = 141 mM, $[Fe^{2+}]_0$ = 1.0 mM, $[Fe^{3+}]_0$ = 1.0 mM and initial pH 3.0 and precipitation pH 11.0. The error bars in the figure indicate the standard deviations which were calculated by replicate data.

3.6. *Effect of temperature*

Temperature is one of the important factors influencing oxidation reaction. In order to study the effect of reaction temperature on the FR-HP process, a series of experiments were conducted at 25, 40 and 50 °C and the results are shown in Fig. 7. The results show clearly that the Ni(II) removal efficiency increases with the increasing temperature. As increasing the temperature from 25 to 50 °C, the Ni(II) removal efficiency increased from 72.1% to 97.2% for Fenton and from 74.3% to 96.7% for Fenton-like system at 30 min. This is because that the reaction of Fenton and Fenton-like could be accelerated by increasing temperature. A higher temperature increased the reaction between hydrogen peroxide and Fe^{3+}/Fe^{2+}, and improved the generation rate of $^{\bullet}OH$ radical [27] and hence enhanced the removal of Ni(II).

Moreover, it can be seen that at 40 and 50 °C, the Ni(II) removal efficiency increased very fast at first 20 min and the increase is only marginal on increasing the temperature further. The Ni(II) removal efficiency is quite similar at 40 and 50 °C after 20 min of reaction, with values around 98%.

3.7. *Residual Fe concentration*

In our research, 1 mM, i.e. 56 mg/l Fe ions was added into the solution, which is well above the European Union directives that allow only 2 mg/l of Fe ions in treated water to be dumped directly into the environment [28]. Therefore, the residual total Fe concentrations were also determined by AAS after FR-HP process. Results indicated that the residual total Fe concentrations were all below the detection limit 0.03 mg/l of total Fe by AAS. Therefore, the solution can be directly discharged in the environment after FR-HP process.

4. Conclusions

FR-HP processes can effectively remove Ni(II) from NiEDTA wastewater. The Fenton-like process presenting higher Ni(II) removal efficiency than Fenton process can be attributed to the mechanism of ligand exchange. For the two systems, Ni(II) removal depends on initial concentrations of Fe ions, H_2O_2 and initial and precipitation pH value. At optimal operation conditions ($[H_2O_2]_0$ = 141 mM, $[Fe^{2+}]_0$ = 1.0 mM, $[Fe^{3+}]_0$ = 1.0 mM, initial pH 3.0 and precipitation pH 11.0), the removal efficiency of Ni(II) were above 92% for the two systems. And temperature has influence on overall nickel removal in the range of 25–50 °C.

Acknowledgements

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