

# Study on the purification property of pyrite and its spectra on the processing of metal-bearing wastewater

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**Abstract** The purification property of pyrite was discussed by using in situ attenuated total reflection-Fourier transform infrared spectroscopy. Results showed that there might be dissolution–adsorption precipitation equilibrium of heavy metals on the surface of pyrite, which is dependent on the surface oxidation of pyrite and the neutralization reaction of carbonate within pyrite. If there was excessive carbonate within pyrite, the “dissolution” of metals would be less than that of the “adsorption precipitation,” making pyrite exhibit its purification property. Based on this property, pyrite was used to process simulated wastewater containing  $Pb^{2+}$ ,  $Hg^{2+}$ ,  $Cd^{2+}$ ,  $Cr(VI)$  and  $Cu^{2+}$ . Results showed that the efficiencies of metal removal exceeded 96%. In addition, reflectance spectroscopy and absorption spectroscopy were also utilized to investigate the simulated metal-bearing wastewater treatment process. Analysis by diffused reflectance infrared Fourier transform spectroscopy confirmed that the superficial hydroxyl groups in pyrite reacted with metal ions

during the wastewater treatment process. Reflectance spectroscopy in the visible region was used to characterize the variation in particle size and specific surface area of pyrite during the wastewater treatment process, which explained its increasing activity when reutilized. Further, analysis by absorption spectroscopy and X-ray photoelectron spectroscopy indicated that the process involved when using pyrite for the treatment of  $Cr(VI)$ -containing wastewater was an adsorption–precipitation process.

**Keywords** Pyrite · Purification property · Heavy metal · ATR-FTIR · DRIFTS · Wastewater treatment

## Introduction

Pyrite ( $FeS_2$ ) is a major mining waste product and natural sulfur mineral as well (Lin and Huang 2008). Since sulfur on its surface rapidly oxidizes when aerated, the sulfate formed would result in acid mine drainage (Nyavor et al. 1996). During the process of surface oxidation and subsequent dissolution of pyrite, some dissolved heavy metals would be released, leading to heavy metal contamination in water and soil. Therefore, the surface oxidation of pyrite has been studied extensively with respect to its role in the production of acid mine drainage. Nonetheless, some related studies mainly focused on the “environmental pollution property” of pyrite (Buckley and Woods 1985; Moses et al. 1987; Sracek et al. 2006; Espana et al. 2007).

The soluble fraction of pyrite is gradually stripped by surface oxidation and dissolution, which results in the release of  $S^{2-}$ , ferrous, ferric, and some other heavy metals into the environment. The  $S^{2-}$  ions will combine with metal ions to form insoluble sulfide. With the decrease in environmental acidity, ferric ions will generate flocculation and

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precipitation, where the hydrolysis precipitation of ferric occurs at the pH 3. This precipitation will in turn stimulate the adsorption precipitation of heavy metals. Therefore, when the environmental conditions change, it is possible for pyrite to exhibit relative purification property. However, few studies have examined and addressed this issue.

Iron sulfide minerals are widely used for the removal of metal ions from wastewater due to their extensive sources, low cost, good removal efficiency, and no secondary pollution, among other advantages. Some studies on the use of iron sulfide minerals for the processing of metal-bearing wastewater were conducted in the 1970s (Brown et al. 1979; Jean and Bancroft 1986). These investigated the adsorption–desorption process of metal ions and minerals by using X-ray photoelectron spectroscopy (XPS) (Hyland et al. 1990; Knipe et al. 1995). In China, however, such studies started to be gradually performed only in the 1990s (Guo et al. 1999; Chen et al. 1999; Lu et al. 2000). Mechanism studies by XPS (Jia et al. 1999) on the minerals used for the processing of metal-bearing wastewater are mainly concerned with confirming the forms of metals adsorbed on mineral surface.

Carbonates are widely distributed in nature and are always found in pyrite. Zhang (Zhang et al. 2007) studied their effect in the thallium-releasing process inside pyrite. Based on the results of aforementioned study, the effect of the environmental chemistry property of pyrite on the migrating and releasing activity of heavy metal was further discussed in this paper. In addition, the removal efficiency of pyrite for the processing of metal-bearing wastewater was tested. However, the use of minerals for the processing of metal-bearing wastewater is a complex process, and the information that can be obtained from a characterization method such as XPS is not adequate. Thus, in this paper, the metal-bearing wastewater treatment process was also investigated on the basis of reflectance spectroscopy and absorption spectroscopy technology. Further, the surface reaction of pyrite and its interaction of pyrite with metal ions were examined.

## Materials and methods

### Reagents and samples

Pyrite samples were collected from pyrite deposits in Yun-fu City, Guangdong Province, China. All samples were

naturally air-dried, then filtrated by a 0.074-mm nylon mesh after being triturated by agate mortar. Elemental compositions of pyrite sample were identified by X-ray Diffraction (XRD), and the results are shown in Table 1. Wastewater containing  $\text{Pb}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$ , and  $\text{Cr(VI)}$  was a simulated acid wastewater and was formed in routine method by using chemical reagents. All reagents were analytically pure, and all solutions were prepared in distilled water.

### Apparatus

An ELAN 6100 ICP-MS instrument (Perkin–Elmer SCIEX, Thornhill, Ontario, Canada), which is equipped with a dynamic reaction cell (DRC) and a Perkin–Elmer Model AS93F auto-sampler, was applied to determine the concentrations of heavy metals in wastewater.

TENSOR-27 FTIR spectrometer (Bruker, Germany), an infrared spectroscopy instrument, was used in the experiment with the following attributes: DTGS detector, resolution of  $4\text{ cm}^{-1}$ ,  $32\times$  scanning capability, and in situ attenuated total reflection technology characterization.

Diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) for surface analysis was performed by the EQUINOX-55 infrared spectrometer and diffuse reflectance accessories. The reflectance spectra in the visible region were measured by a reflectance spectrometer equipped with optical fiber conduction (Zhang and Chen 2006). Absorption spectrum was measured by 721-spectrophotometer. XPS spectrum was provided by ESCA-LBMK-II X-ray Photoelectron Spectrometer.

### Experimental procedures of pyrite used for processing of metal-bearing wastewater

- Step 1 The pyrite powder used for the experiment was  $<0.074\text{ mm}$ . The pH of the simulated wastewater containing  $\text{Pb}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$ , and  $\text{Cr(VI)}$  was controlled in the range of 1–2. The powder was added into the wastewater in the proportion of 1:10. Then the wastewater was stirred for 60 min at room temperature.
- Step 2 The solution obtained from step 1 was deposited in static for 5 h. Then the clear solution above the precipitate was discharged.
- Step 3 The concentrations of the metals in clear solution were determined by ICP-MS.

**Table 1** Elemental compositions of pyrite sample (%)

Sample	Pyrite	Hematite	Magnetite	Limonite	Silicate	Carbonate	Clay minerals	Gypsum
Pyrite	88	ND	ND	1	3	5	2	ND

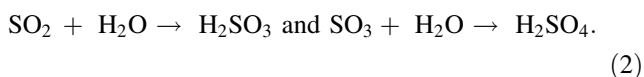
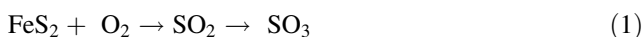
ND not detected

## Results and discussion

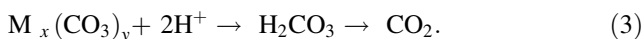
### The purification property of pyrite

Surface oxidation and acidification of pyrite may dissolve its soluble fraction, which results in the release of a proportional amount of heavy metal. The surface dissolution and surface acidification of pyrite could be validated by the following experiment.

A small amount of pyrite powder sample was placed on the surface of ATR crystalloid. If the surface dissolution and surface acidification of pyrite occur in the presence of oxygen and vapor in the air, then the following reaction could be expected:



Further, the carbonates within pyrite could react with  $\text{H}_2\text{SO}_4$  and  $\text{H}_2\text{SO}_3$  to release  $\text{CO}_2$ :

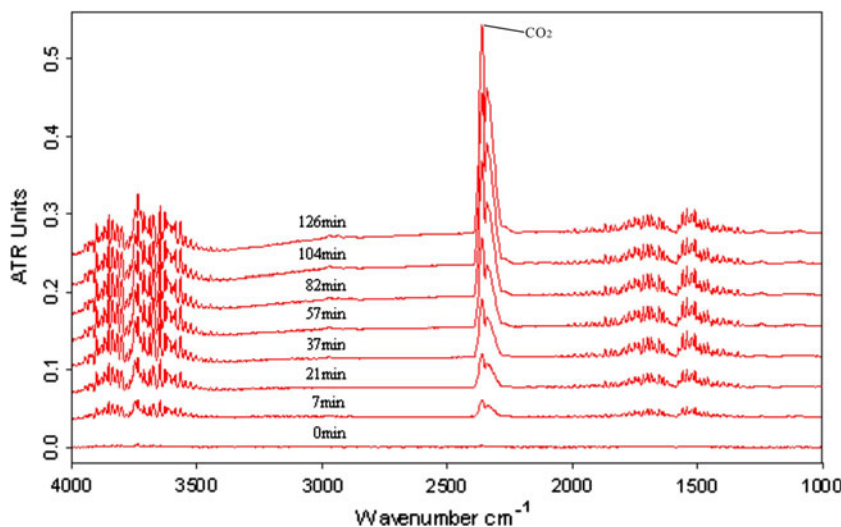


The release of  $\text{CO}_2$  is retained in the lacuna of the sample during the reaction. The concentration of  $\text{CO}_2$  increased as the reaction progressed, as indicated by ATR infrared spectroscopy. The ATR infrared spectroscopy of pyrite under a temperature of  $28^\circ\text{C}$  and a relative humidity of 80% are shown in Fig. 1. As shown in the same figure, the double-absorption peaks of  $\text{CO}_2$  at  $2,360$  and  $2,340\text{ cm}^{-1}$  were enhanced gradually with time extension, which validates the surface dissolution and surface acidification of pyrite.

Surface dissolution occurs on the surface of pyrite, and this dissolution includes the formation of  $\text{H}^+$  or surface

acidification. Lu (Lu et al. 2005) found that after the surface acidification of pyrite, an acid liquid membrane forms on its surface. This can be proven to some extent by the absorption evident near the spectral range of  $3,950\text{--}3,500\text{ cm}^{-1}$  and  $1,800\text{--}1,400\text{ cm}^{-1}$  (Fig. 1), wherein the absorption should be caused by the intermediate species generated in the process of surface oxidation and acidification of pyrite in the presence of water vapor. Whether the heavy metal released from pyrite might penetrate the acid liquid membrane or be adsorbed on the surface of pyrite depends on the acidity of the liquid membrane, the concentration of  $\text{H}^+$ . The higher the acidity, the greater the capacity of  $\text{H}^+$ 's exchange adsorption of heavy metal from the surface of pyrite into the liquid membrane, resulting in a greater migration activity of heavy metal. On the other hand, surface oxidation of pyrite might accelerate the acidity of the liquid membrane. However, in situ infrared spectroscopy proved that the carbonates within pyrite (Table 1) could react with the surface oxidation product, sulfuric acid, which would restrict the acidity of the liquid membrane (Fig. 1). The reduction in acidity would stimulate the released heavy metal ions to combine with  $\text{S}^{2-}$  ions and form insoluble sulfides, or be directly adsorbed on the surface of pyrite. Hence, the carbonate stimulates the adsorption–precipitation of heavy metal on the surface of pyrite since it could increase the pH of liquid membrane. This proves the dissolution–adsorption precipitation equilibrium of heavy metal on the pyrite surface when under the double effects of oxidation of pyrite and the neutralization of carbonate. If there was excess carbonate, then there would be lesser dissolution of heavy metal than adsorption precipitation. This would result in the exhibition of the purification property. On the contrary, pyrite would also exhibit its environmental pollution property, which leads to acid mine drainage and heavy metal pollution.

**Fig. 1** In situ ATR-FTIR spectra of pyrite in the air at different time. (temperature,  $25^\circ\text{C}$ ; relative humidity, 85%)



### Removal efficiencies of using pyrite for processing of metal-bearing wastewater

Using the purification property of pyrite, its efficiency on the removal of metal ions from simulated wastewater was tested. The results are shown in Table 2. When pyrite was used to remove metal ions such as  $\text{Pb}^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$ , and Cr(VI) from simulated wastewater, they presented satisfactory removal efficiencies within the range of 96–99%. The results obtained were in accordance with those reported by Zhang (Zhang and Chen 2005). When the concentration of  $\text{Pb}^{2+}$ , Cr(VI) and  $\text{Cu}^{2+}$  in wastewater is 100 mg/L for all ions, it could meet the discharge standards after treatment (the highest discharge concentrations for first-category pollutants are  $C_{\text{Pb}} \leq 1.0$  mg/L,  $C_{\text{Cr}} \leq 1.5$  mg/L; the highest discharge concentration for second-category pollutants is primary standard  $C_{\text{Cu}} \leq 0.5$  mg/L). When the concentration for both  $\text{Hg}^{2+}$  and  $\text{Cd}^{2+}$  is 10 mg/L, it could meet the discharge standards after treatment (the highest discharge concentrations for first-category pollutants are  $C_{\text{Cd}} \leq 0.1$  mg/L,  $C_{\text{Hg}} \leq 0.05$  mg/L). In addition, the pH of after-treatment solution approaches a neutral level without addition of alkali due to the neutralization effect of carbonate within the pyrite. This indicates that the carbonate within the pyrite is a form of natural purification regulator.

The removal of metal ions using pyrite showed satisfactory results. This was determined by pyrite's own chemical property. On one aspect, under a certain acidic condition, the dissociation of pyrite tends to release dissolved  $\text{S}^{2-}$  ions which will combine with metal ions and form insoluble sulfide. On another aspect, as the acidity decreases during wastewater treatment process, the dissolved iron and aluminum ions generate flocculation and precipitation. The aforementioned processes are always accompanied by strong adsorption, which can completely precipitate the metal ions.

### Investigation performed by DRIFTS on pyrite used for metal-bearing wastewater treatment

Investigating pyrite powder by transmission infrared Fourier transform spectroscopy proved to be difficult due to its opaqueness. The few studies available (Peng and Liu 1982; Lanigan and Pidosny 2007) only provided that the infrared spectrum with wave number is below  $1,600 \text{ cm}^{-1}$ , and the spectrum given is simple and in the spectral range of  $1,600\text{--}500 \text{ cm}^{-1}$  only when there is a weak absorption near  $1,400$  and  $1,100 \text{ cm}^{-1}$ . DRIFTS established on scattering and absorption is suitable for opaque solid and its surface analysis (Moradi et al. 1999; Zhang et al. 2003). Therefore, DRIFTS was chosen for the investigation of metal-bearing wastewater treatment. The resulting DRIFTS spectrum of pyrite is shown in Fig. 2. The absorption peaks at  $3,350$  and  $3,401 \text{ cm}^{-1}$  are attributed to  $\text{-OH}$  stretching vibration, which indicates the abundance of superficial hydroxyl groups in the pyrite. There are also absorption peaks near  $1,400$  and  $1,100 \text{ cm}^{-1}$ , but both are strong absorption peaks compared with the spectra reported by Peng (Peng and Liu 1982). The absorption peaks at  $1,438$ ,  $872$  and  $707 \text{ cm}^{-1}$  associated with  $\text{CO}_3^{2-}$  (Peng and Liu 1982) indicate the presence of carbonates within pyrite, which was further proven by XRD (Table 1). Wu (1994) reported that sulfate had a very strong absorption band in the spectral region between  $1,130$  and  $1,080 \text{ cm}^{-1}$  in the infrared Fourier transform spectroscopy spectra. Thus, the strong absorption peak near  $1,152 \text{ cm}^{-1}$  associated with the stretching vibration of  $\text{SO}_4^{2-}$  indicates that the oxidizing reaction of pyrite is accompanied by sulfate formation. The study performed by DRIFTS shows that the pyrite used in the experiment was "impure".

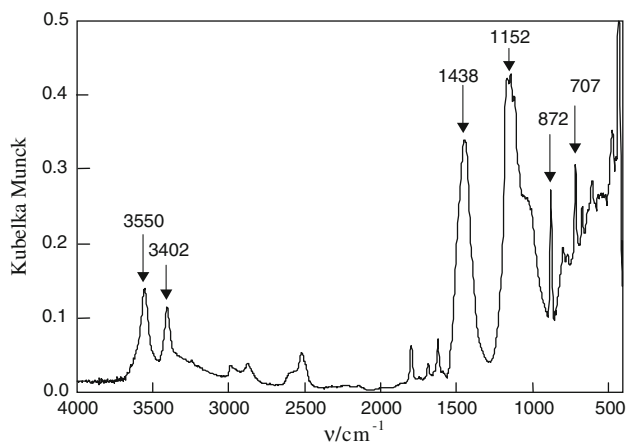
The DRIFTS spectra of pyrite in the hydroxyl group region are shown in Fig. 3, in which *a* is the spectrum of pyrite before being used for the processing of acid metal-bearing wastewater, and *b* to *d* is the spectra of pyrite after

**Table 2** Results of using pyrite for simulated acid metal-bearing wastewater treatment (mg/L)

Heavy metal ions	$\text{Pb}^{2+}$	$\text{Cd}^{2+}$	$\text{Hg}^{2+}$	$\text{Cu}^{2+}$	Total Cr <sup>a</sup>	pH value
Before treatment $C_M$	1.0	1.0	1.0	1.0	1.0	1.80
After treatment $C_M$	0.012	0.036	0.019	0.008	0.025	6.30
Removal rate (%)	98.8	96.4	98.1	99.2	97.5	–
Before treatment $C_M$	10.0	10.0	10.0	10.0	10.0	1.84
After treatment $C_M$	0.022	0.091	0.031	0.018	0.035	6.35
Removal rate (%)	99.8	99.1	98.7	99.8	99.6	–
Before treatment $C_M$	50.0	50.0	50.0	50.0	50.0	–
After treatment $C_M$	0.122	0.787	0.285	0.101	0.750	6.48
Removal rate (%)	99.8	98.4	99.4	99.8	98.5	–
Before treatment $C_M$	100.0	100.0	100.0	100.0	100.0	1.46
After treatment $C_M$	0.312	1.685	0.455	0.111	1.235	6.51
Removal rate (%)	99.7	98.3	99.5	99.8	98.5	–

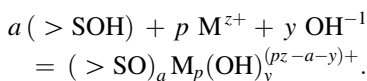
$C_M$  the concentration of metals in wastewater

<sup>a</sup> Total Cr: including  $\text{Cr}^{3+}$  and Cr(VI)

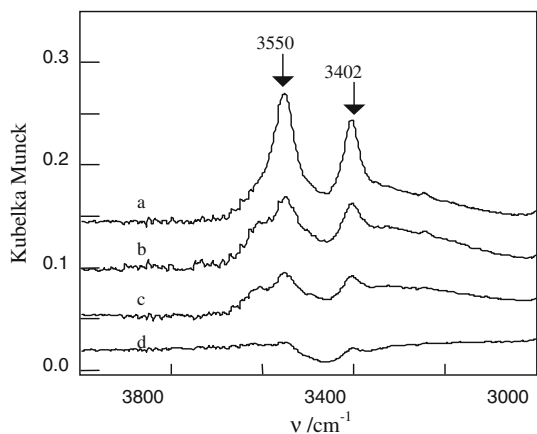


**Fig. 2** DRIFTS spectrum of pyrite

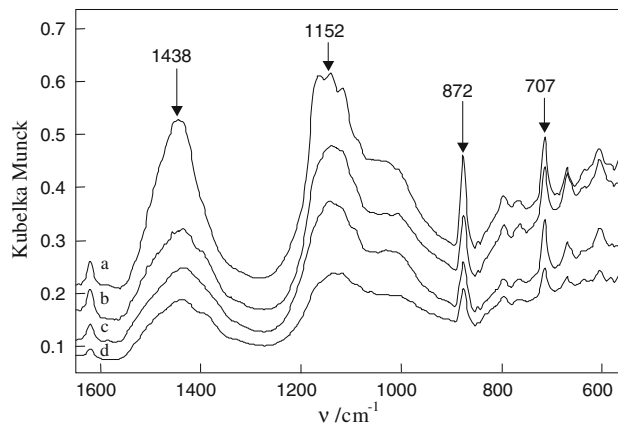
being used for the processing of acid metal-bearing wastewater in concentrations of 10, 50, and 100 mg/L, respectively. As shown in Fig. 3, with the increase in metal concentrations in wastewater treatment, absorption peaks connected with the superficial hydroxyl groups in pyrite gradually decreased until they completely collapsed (Fig. 3d). This indicates that the metal ions reacted with the superficial hydroxyl groups in pyrite and accelerated the adsorption precipitation of these metal ions. This reaction on mineral surface is common and widely ascribed to the adsorption of metal ions by minerals. The reaction equation can be described as follows (Davis and Kent 1990):



In the reaction equation, the parameters of *a*, *p*, and so on will vary with the type of ions and the pH of the solution.



**Fig. 3** DRIFTS spectra of pyrite in the -OH region. *a* Before being used for the processing of metal-bearing wastewater; *b–d* after being used for the processing of acid metal-bearing wastewater in concentrations of 10, 50, and 100 mg/L, respectively

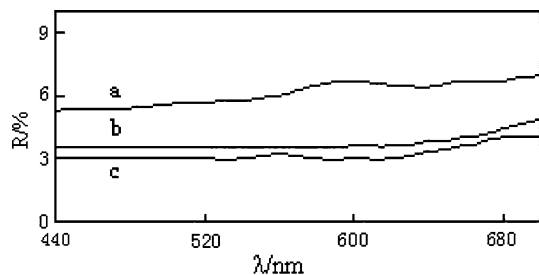


**Fig. 4** DRIFTS spectra of pyrite in the spectral region between 1,650 and 550  $cm^{-1}$ . *a* Before being used for the processing of metal-bearing wastewater; *b–d* after being used for the processing of acid metal-bearing wastewater in concentrations of 10, 50, and 100 mg/L, respectively

Figure 4 shows the DRIFTS spectra of pyrite in the spectral region between 1,650 and 550  $cm^{-1}$ , in which *a* is the spectrum of pyrite before it was used for the processing of acid metal-bearing wastewater, and *b* to *d* is the spectra of pyrite after it was used for the processing of acid metal-bearing wastewater in concentrations of 10, 50, and 100 mg/L, respectively. As shown in Fig. 4, as compared to the blank pyrite (Fig. 4a) the consumed pyrite showed decreasing absorption peaks at 1,438, 1,152, 872, and 707  $cm^{-1}$ . The decrease in absorption peak at 1,152  $cm^{-1}$  indicated that a part of the sulfate attached to the pyrite surface was dissolved. The decrease in absorption peaks at 1,438, 872, and 707  $cm^{-1}$  showed that the reaction of carbonates with  $H^+$  reduced the acidity of the solution, which explains why the pH approached neutrality ( $pH_e \approx 7$ , Table 2) without additional alkali as the treatment progressed. This is a natural alkalization process, which is beneficial to the flocculation adsorption of iron and aluminum, because it can completely precipitate metal ions.

Investigation performed by reflectance spectroscopy on pyrite being used for processing of metal-bearing wastewater

Reflectance spectroscopy applied in this experiment provided significant results. The reflectance spectra in the visible region of pyrite before and after utilization were measured by a reflectance spectrometer equipped with optical fiber conduction. The results presented in Fig. 5 are the spectra of pyrite before treatment and after being used to process acidic distilled water or acid metal-bearing wastewater, which are denoted as *a*, *b*, and *c*, respectively. As shown in Fig. 5, the reflectivity of pyrite after treating acidic distilled water (Fig. 5b) and acid metal-bearing

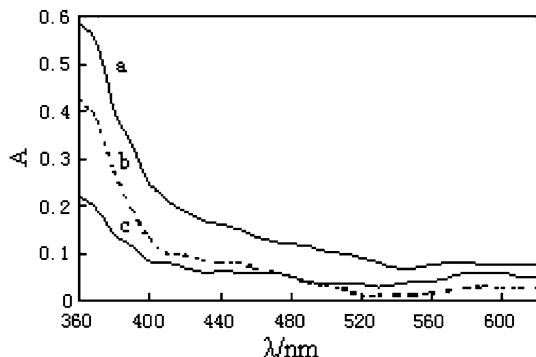


**Fig. 5** Reflection spectra in the visible region of pyrite. *a* Before treatment; *b–c* after being used to process acidic distilled water or metal-bearing wastewater, respectively

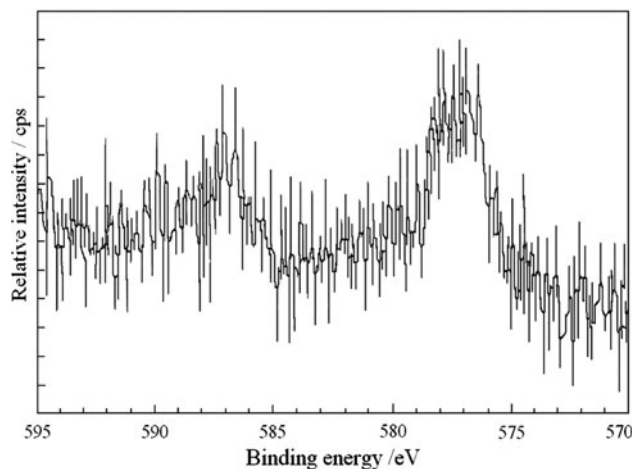
wastewater (Fig. 5c) significantly decreased as compared to that before being used for the treatment process (Fig. 5a). Evident dissolution occurred in pyrite, which lessened its particle size but increased its specific surface area. The increase in specific surface area further promoted the adsorption of pyrite. This may partially explain the increasing activity of pyrite when it was reutilized for metal-bearing wastewater treatment.

Investigation performed by absorption spectroscopy on pyrite being used for processing of Cr(VI)-containing wastewater

The major step in the process of metal-bearing wastewater treatment by using pyrite is the combination of  $S^{2-}$  ions with metal ions and the formation of insoluble sulfide. However,  $S^{2-}$  ions are impossible to combine with  $Cr_2O_7^{2-}$  and form insoluble sulfide from the chemical point of view. Some useful information was obtained by investigating the absorption spectra of a  $Cr_2O_7^{2-}$ -containing solution after treatment with pyrite as shown in Fig. 6. The absorption curves measured after pyrite was utilized to treat  $Cr_2O_7^{2-}$ -containing wastewater for 0, 10 and 20 min are shown in *a*, *b* and *c*, respectively. The absorption near



**Fig. 6** Absorption spectra in the visible region of a  $Cr_2O_7^{2-}$ -containing solution after treatment with pyrite at different time. *a* 0 min; *b* 10 min; *c* 20 min)



**Fig. 7** XPS spectrum pattern of  $Cr_{2p}$  on the surface of pyrite

360 nm evidently decreased with the extension of treatment time, which indicated the rapid reduction in concentration of  $Cr_2O_7^{2-}$ . Further, the incidence of a higher absorption in the region between 560 and 650 nm in the *c* curve than that in the *b* curve indicated that there was  $Cr^{3+}$  formation ( $Cr^{3+}$  has a weak absorption band in the region between 560 and 650 nm). Consequently, the incidence of a reduction process from Cr(VI) to  $Cr^{3+}$  in the process of using pyrite for the  $Cr_2O_7^{2-}$ -containing wastewater treatment was proven by absorption spectroscopy analysis. In addition, pyrite was analyzed by XPS. As shown in Fig. 7, the electron binding energy near 577 and 587 eV was associated with the  $Cr_{2p_{3/2}}$  and  $Cr_{2p_{1/2}}$  of  $Cr(OH)_3$ , respectively, which indicated the existence of  $Cr(OH)_3$  species. Hence, in the process of  $Cr_2O_7^{2-}$ -containing acid wastewater treatment, a redox reaction occurred between Cr(VI) and the dissolved  $Fe^{2+}$  and  $S^{2-}$ . Then, there is formation of  $Cr^{3+}$  which is in the form of  $Cr(OH)_3$  adsorbed by pyrite or as a direct precipitate.

## Conclusion

The purification property of pyrite was investigated using in situ ATR-FTIR spectroscopy in this paper. This property was characterized by pyrite's own chemical property and the carbonates it contained in the pyrite. Further, based on its purification property, pyrite was utilized to remove metal ions such as  $Pb^{2+}$ ,  $Hg^{2+}$ ,  $Cd^{2+}$ ,  $Cu^{2+}$ , and Cr(VI) from simulated acid wastewater. Experimental results indicated that the efficiencies of metal removal exceeded 96% in different concentrations of the aforementioned metal ions.

In addition, using pyrite for the processing of simulated metal-bearing wastewater was also investigated by applying reflectance spectroscopy and absorption spectroscopy. The experimental results are as follow:

1. Analysis performed by DRIFTS indicated that the metal ions that reacted with the superficial hydroxyl groups in pyrite accelerated their adsorption precipitation of metal ions during the metal-bearing wastewater treatment process. This explains why the pH approached neutrality without additional alkali in the after-treatment solution. This was a natural alkalization process performed by the carbonates within pyrite. Further, investigation on the reflectance spectra in the visible region indicated that particle size of pyrite became smaller, and its specific surface area became larger during treatment, which may partially explain the increasing activity of pyrite after it was reutilized.
2. Analysis performed through absorption spectroscopy and XPS showed that during the processing of Cr(VI)-containing acid wastewater, an adsorption precipitation process from Cr(VI) to Cr<sup>3+</sup> then to Cr(OH)<sub>3</sub> occurred.

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