

## Research Article

# The Effect of Proton and Arsenic Concentration on As(III) Removal by Hematite and Kaolin Complexes

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With the intensification of human activities, arsenite (As(III)) pollutant from the soil and ground water has been a threat to human health, and the problem gradually becomes the focus of attention. In this study, the effects of several environment factors on As(III) removal ability of complex minerals are determined through the analysis of mineralogical characteristics of the complex synthesis of hematite and kaolin, using X-ray diffraction, Fourier transform infrared, and specific surface area. In the results, the XRD patterns of hematite and kaolin complexes indicate that the loading covers up some characteristic peaks of minerals in kaolin, which can be that hematite loading decrease the order of structure for some minerals. With increasing Fe content, the hydroxyl sites gradually increase, therefore strengthening the As(III) removal ability of complexes. With increasing the As(III) concentration, the removal ratios of kaolin on As(III) almost keep unchanged and that of complexes show no obvious regularity. However, with the pH increasing, the removal ratios of all samples keep increasing. Furthermore, the increasing of As(III) concentration and pH both improve As(III) removal amount significantly, especially for As(III) concentration. In addition, there is no forming of new mineral through it as observed by XRD. Therefore, hematite loading can promote the As(III) removal on kaolin through adsorption in different environments, which can provide a better method for the remediation of arsenic pollution.

## 1. Introduction

Arsenic (As), as a nonmetallic element, is one of the common pollutants and increases the risk of cancer [1]. When a large amount of As is released into the soil and water environments, the ecological environment and human health are threatened extremely [2–4]. For example, 30 million people in Bangladesh were affected by the As pollution in groundwater, in which As concentration is higher than the maximum allowable concentration of drinking water from the World Health Organization [5]. In the soil, most As is inorganic arsenic, existing as in trivalent arsenic (As(III)) and pentavalent arsenic (As(V)), and As(III) holds the stronger toxicity and easier migration than As(V) [6–9]. Therefore, the adsorption is an effective and economic technique for the remediation of the As(III) pollution, which is widely adopted [10–12]. Many studies on the removal of As(III) by single

pure phase mineral has been reported, but that by binary or ternary complex needs to be further studied, which can provide a better adsorbent for the remediation of the As(III) pollution.

In the natural soil environment, As is usually adsorbed and/or oxidized by silicate minerals and/or metal oxides, which is the key process to control the effective concentration of As [13–15]. Thus, the soil minerals usually act as a natural adsorbent for different pollutions [16]. Therein, the high content of silicate minerals, which are widely distributed, is characterized by good chemical stability, high cation/anion exchange, and good adsorption ability [17]. Additionally, their surface reactivity shows significant influence on controlling the concentration of contaminants in soil and water. Among them, kaolin clay is the most abundant mineral, which consists of kaolinite with other impurities such as illites, smectites, feldspars, and quartz [18].

Kaolin clay with negative charge can adsorb heavy metals with positive charge through ionic exchange with  $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{K}^+$ , and/or  $\text{Mg}^{2+}$ , which displays a good potential for contaminant removal [19], and acts as a low-cost adsorbent with metal binding capacity [20]. However, kaolin clay holds a relatively weaker adsorption ability for anion, such as arsenate and phosphate. Therefore, the modification of adsorption ability of kaolin clay on anion is still required.

In the natural environment, silicate minerals do not exist alone and usually form complexes with metal oxides and/or organic, leading to the change of the physical-chemical properties of minerals, further the migration and transformation ability of pollutants [21, 22]. Many studies about the modification of kaolinite have been reported. The modification are mainly performed through intercalation and surface loading compounds, forming complexes and bringing out a better adsorption ability of the minerals on As [23, 24]. In the tropical or subtropical regions, hematite is a common and important active mineral in soils and can form weakly crystalline iron plaque on other soil minerals, which would greatly improve the adsorption ability of other soil minerals. Furthermore, hematite is proved to be an effective adsorbent to remove arsenate from drink water [25, 26]. Therefore, in this study, the characteristic of hematite loaded on kaolin clay complexes will be determined, and the interaction between the complexes and As(III) was explored further to explore the important of natural complex minerals. Furthermore, the effects of As(III) concentration and pH on that interaction are investigated. Especially for pH, it is a very important factor to the adsorption. Many research shows that the adsorptions of minerals on As(III) are decreasing with pH increasing just like Figure 1 [27–29]. All these results will provide contribution for us to understand the migration and transformation of As(III) in red soil.

## 2. Materials and Methods

**2.1. Sample Synthesis.** 125 mL deionized water was heated to 90°C, and then, 10 g  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  was added and still completely dissolved. Next, 1 g or 3 g kaolin was added, followed by the addition of 75 mL KOH (1 mol/L) solution to obtain the brown precipitate. Finally, 12.5 mL of 1 mol/L  $\text{NaHCO}_3$  was added to the above solution and the mixed solution was kept at 90°C for 48 h. During this process, it was kept as sealed. The final red precipitate was centrifuged and cleaned with deionized water until the conductivity was less than 20  $\mu\text{S}/\text{cm}$ , then freeze-dried, and stored in a closed drying environment for standby. The complexes are named as He@kaolin-1 and He@kaolin-3, respectively.

**2.2. Characterization of Samples.** The obtained samples were characterized by the Bruker D8 Advance powder X-ray diffractometer (XRD) using  $\text{Cu K}\alpha$  radiation ( $\lambda = 0.15418$  nm), and the measuring conditions were 40 kV and 40 mA. The specific surface area (SSA) was examined at 110°C for 3 h under vacuum prior by nitrogen adsorption-desorption isotherms at liquid nitrogen temperatures according to Brunauer-Emmett-Teller (BET) model using

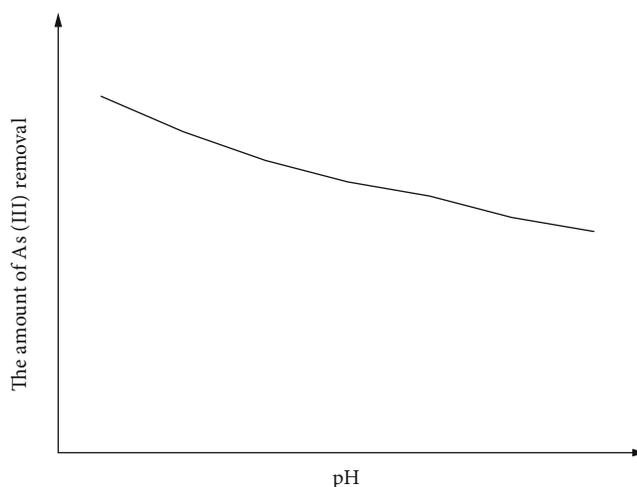


FIGURE 1: As removal in pH.

Micromeritics ASAP 2020. Fourier transform infrared (FTIR) spectra were acquired using the Bruker VERTEX 70 FTIR spectrometer (64 scans,  $4\text{ cm}^{-1}$  resolution).

**2.3. Removal of As(III) by Minerals and Complexes at Different Initial Concentrations.** At room temperature, 0.010 g sample was accurately weighed and added into 50 mL of 6 mg/L, 12 mg/L, and 24 mg/L As(III) solution with 0.1 mol/L  $\text{NaNO}_3$ . The pH was kept  $6 \pm 0.05$  during the process and magnetic stirring reaction for 300 min. Finally, the suspension was filtered with 0.22  $\mu\text{m}$  microporous membrane, and the As(III) concentration was determined by atomic fluorescence spectrometer (AFS-8220, Beijing Jitian Analytical Instrument, China).

**2.4. Removal of As(III) by Minerals and Complexes at Different Initial pH.** At room temperature, 0.010 g sample was accurately weighed and added into 50 mL of 6 mg/L As(III) solution with 0.1 mol/L  $\text{NaNO}_3$ , and the pH was, respectively, kept  $4 \pm 0.05$ ,  $6 \pm 0.05$ , and  $8 \pm 0.05$  during the process and magnetic stirring reaction for 300 min. Finally, the suspension was filtered with 0.22  $\mu\text{m}$  microporous membrane, and As(III) concentration was determined by Atomic fluorescence spectrometer (AFS-8220, Beijing Jitian Analytical Instrument, China).

## 3. Results

**3.1. Analysis of X-Ray Diffraction.** The XRD patterns of kaolin and complexes samples were shown in Figure 2. In the XRD pattern of kaolinite, three peaks of diamond labeling represent the presence of kaolinite. There are other minerals in the kaolin along with kaolinite. However, in the XRD pattern of He@kaolin-3, the peaks of kaolin are almost disappeared, and some new characteristic peaks are appeared. These peaks were consistent with (012), (104), (110), (113), (024), and (116) crystal planes of hematite in JCPDS card (24-0072) [30, 31]. With increasing the Fe content, the peak intensity of hematite strengthens in the XRD pattern of He@kaolin-1. However, the content of kaolin is lower than

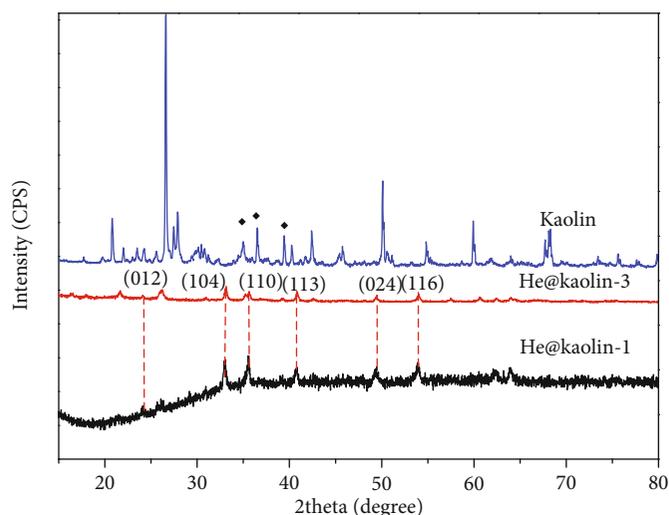


FIGURE 2: XRD patterns of kaolin, He@kaolin-3, and He@kaolin-1.

that of hematite leading to the appearance of characteristic peaks of kaolin in He@kaolin-1 and He@kaolin-3, which will be further proved later.

**3.2. Analysis of Fourier Transform Infrared Spectroscopy.** The FTIR patterns of kaolin and complexes samples were shown in Figure 3. In the three FTIR patterns, the bands at 3430 and 1630  $\text{cm}^{-1}$  represent the  $-\text{OH}$  stretching vibration in adsorbed water molecule [32]. The bands at 1090, 895, and 822  $\text{cm}^{-1}$  represent the vibration of Si-O-Si/Al, Si-OH, and Al-OH, respectively [24]. Moreover, the bands at 573 and 473  $\text{cm}^{-1}$  in kaolin can be ascribed to Si-O-Al/Fe and Si-O-Si, but that at near wavenumber in He@kaolin-1 and He@kaolin-3 can be ascribed to Fe-O-Fe. Further, the new adsorbed peak at 1345  $\text{cm}^{-1}$  is the vibration of Fe-OH. Therefore, with Fe content increasing, the contents of both  $\text{H}_2\text{O}$  and  $-\text{OH}$  increase.

**3.3. Analysis of Specific Surface Areas.** The SSA of kaolin and complexes samples were measured using BET method, and the adsorption and desorption curves of  $\text{N}_2$  were shown in Figure 4. The adsorption and desorption curves (Figure 4(a)) and pore size distribution curves (Figure 4(b)) of kaolin and He@kaolin-3 are similar, especially the position and shape size of hysteresis loop. Thus, the corresponding SSAs were 6.48 and 15.87  $\text{cm}^2/\text{g}$ , and the main pore sizes both range from 20 to 50 nm. But the SSA and main pore size of He@kaolin-1 are 163.43  $\text{cm}^2/\text{g}$  and  $\sim 5$  nm, respectively, which are different from those of the other samples. This phenomenon can be attribution to the important effect of hematite content on the SSA and main pore size of mineral.

**3.4. As(III) Removal by Minerals at Different Initial as(III) Concentrations.** The removal ratios of As(III) by kaolin, He@kaolin-1, and He@kaolin-3 at different initial As(III) concentrations are shown in Figure 5. For three samples, with Fe content increasing, the removal ratios of As(III) significantly increase at different As(III) concentrations of 6, 12, or 24 mg/L. The maximum difference can reach removal

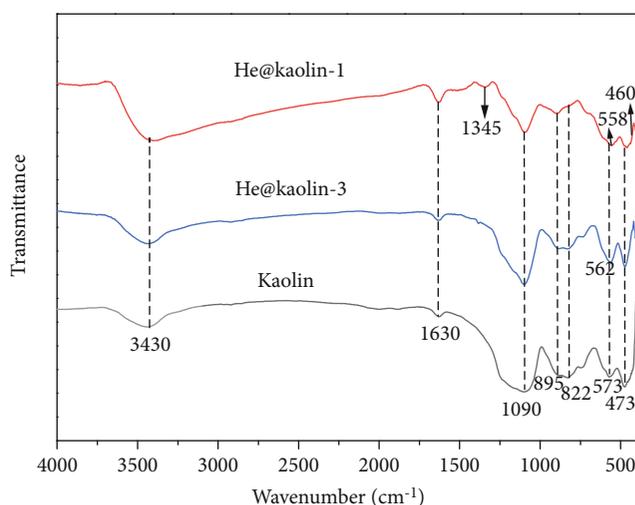


FIGURE 3: FTIR spectra of kaolin, He@kaolin-3, and He@kaolin-1.

ratio of  $\sim 50\%$ . However, the removal ratios of As(III) by the same mineral display a minor regular change. For kaolin and He@kaolin-3, the As(III) removal ratios hardly change, while the removal ratios of As(III) on He@kaolin-1 first increase and then decrease. The reasons are that the increasing concentration of As(III) decreases the removal ratio of As(III) while its adsorption on mineral decrease. In addition, the calculation results showed in Table 1 indicate that the removal amount of As(III) is always increasing significantly with the increasing of Fe content or As(III) concentration.

Furthermore, the adsorption products of kaolin, He@kaolin-1, and He@kaolin-3 at As(III) concentrations of 24 mg/L were analyzed by XRD in Figure 6. Through comparison with the XRD patterns of kaolin, He@kaolin-1, and He@kaolin-3 in Figure 2, the characteristic peaks at  $21^\circ$  and  $26^\circ$  in kaolin always appeared in He@kaolin-1, He@kaolin-3, and their adsorption products, which is consistent with characteristic peaks of quartz. The other peaks were gradually disappearance or weak after adsorbed As(III), indicating that effect of adsorbed As(III) on structural order

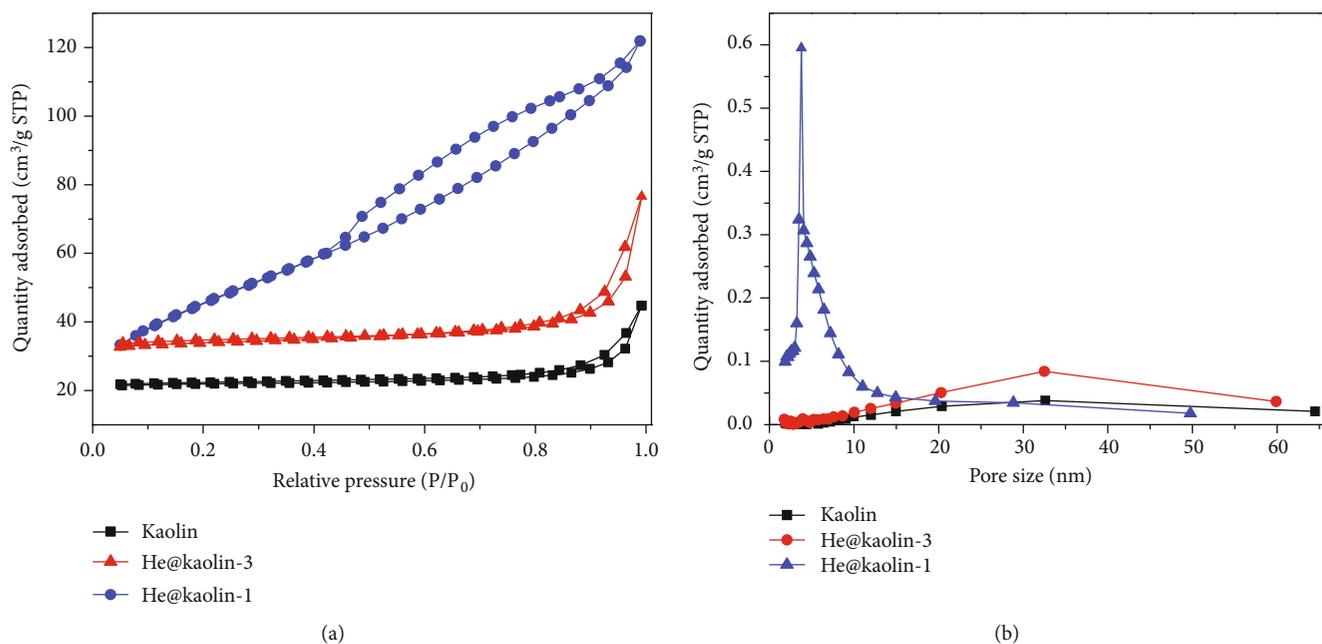


FIGURE 4:  $N_2$  adsorption-desorption isotherm and pore size distribution of kaolin, He@kaolin-3 and He@kaolin-1.

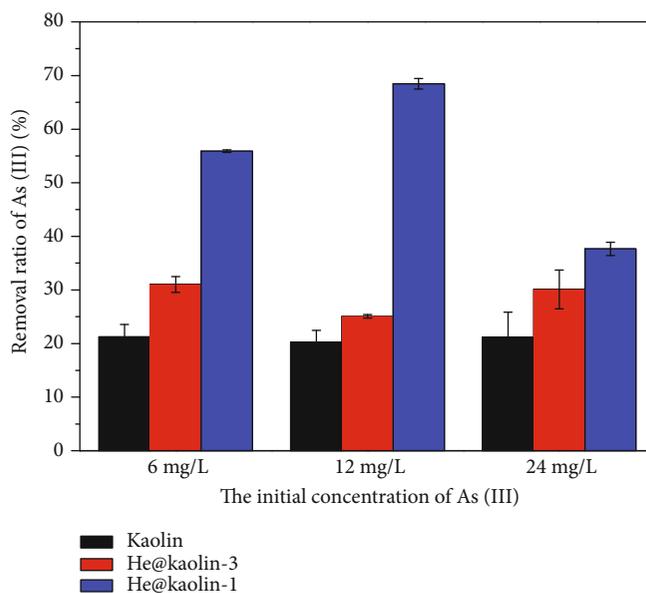


FIGURE 5: Removal ratio of As(III) by kaolin, He@kaolin-1, and He@kaolin-3 at different initial As(III) concentrations (6, 12, and 24 mg/L).

of part minerals in kaolin is strong, like ion adsorption decreasing characteristic peaks of XRD [33, 34]. With increasing the content of Fe, the two characteristic peak intensities of quartz are gradually weak in Figure 2. Meanwhile, As(III) adsorption changed the peak intensity ratio of (104) and (110) in hematite, which is caused by decreasing the peak intensity of (110). The positions of (104) and (110) peaks in hematite for He@kaolin-1 and He@kaolin-3 were obviously shifting to low angle. It indicates that there can be a large amount of As(III) adsorbed on (110) and (104) planes with different influences [35].

TABLE 1: Removal amount of As(III) by kaolin, He@ kaolin-1, and He@ kaolin-3 at different initial As(III) concentrations (6, 12, and 24 mg/L).

Samples	Kaolin (mg/L)	He@kaolin-3 (mg/L)	He@kaolin-1 (mg/L)
6 mg/L	1.28	1.56	3.35
12 mg/L	2.43	3.01	8.22
24 mg/L	5.09	7.22	9.04

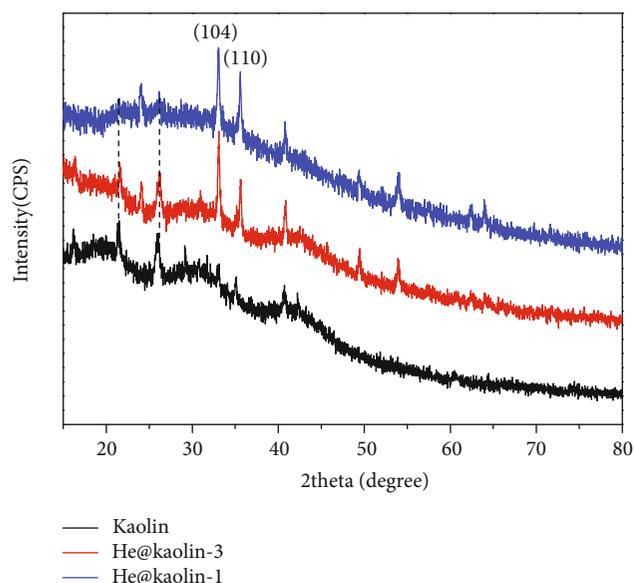


FIGURE 6: XRD patterns of the adsorption products of kaolin, He@kaolin-3, and He@kaolin-1 at 24 mg/L As(III).

Moreover, there were no new mineral appearances in the adsorption products.

**3.5. As(III) Removal by Minerals at Different pH.** Figure 7 shows the results of removal ratio of As(III) by kaolin, He@kaolin-1, and He@kaolin-3 at different pH. With the content of Fe increasing, all removal ratios of As(III) significantly increase at pH of 4, 6, and 8. Especially between kaolin and He@kaolin-1, the differences of removal ratio at different pH can reach ~35%. The removal ratios of As(III) by the same mineral at pH of 4, 6, and 8 are also increased except for kaolin at pH 8, which is not consistent with the results at different concentrations of As(III). Through calculation, the removal amount of As(III) is always significantly increased no matter if the content of Fe or the pH in Table 2 is increasing.

Through analysis of the adsorption products of kaolin, He@kaolin-1, and He@kaolin-3 at different pH (Figure 8), the results of XRD showed that, with pH from 4 to 8, there is almost no changes for kaolin, and the intensities and positions (104) and (110) peaks in hematite for He@kaolin-1 and He@kaolin-3 were obviously increasing and shifting to low angle. Therefore, arsenite can better enter into the structure of hematite at high pH and high concentration of As(III), not only adsorption on the surface of hematite. There were no new mineral appearances in the XRD results of products.

## 4. Discussions

**4.1. The Effect of Hematite Loading on As(III) Removal.** With the ratio of kaolin to hematite decreasing, the characteristic peaks of kaolin are gradually decreasing and disappearance, while that of hematite is increasing. The results indicate that the complex of kaolin and hematite can be synthesized. Through the FTIR analysis, with increasing the content of

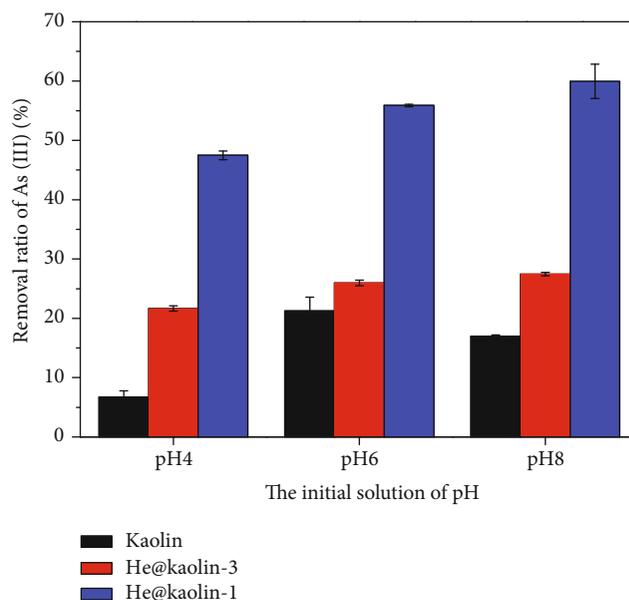


FIGURE 7: Removal ratio of As(III) by kaolin, He@kaolin-1, and He@kaolin-3 at different pH values of 4, 6, and 8.

TABLE 2: Removal amount of As(III) by kaolin, He@kaolin-1, and He@kaolin-3 at different pH of 4, 6, and 8.

Samples	Kaolin (mg/L)	He@kaolin-3 (mg/L)	He@kaolin-1 (mg/L)
pH 4	0.40	1.30	2.85
pH 6	1.28	1.56	3.35
pH 8	1.02	1.65	3.60

hematite, the vibration of Fe-OH and the -OH stretching vibration in the adsorbed water molecule are increasing. In addition, the hematite loading can significantly enhance the SSA, which can be mainly from hematite. Therefore, hematite loading on kaolin can increase the active sites and SSA of the complex, leading to decreasing the electrostatic repulsion and promoting the adsorption of As(III) on complex [36, 37]. For further analysis data, at pH 6, the removal amount of As(III) adds 2.07 mg/L with Fe content increasing (or kaolin concentration decreasing) from kaolin to He@kaolin-1 at As(III) concentration of 6 mg/L, while that with an addition of 3.81 mg/L at As(III) concentration from 6 mg/L to 24 mg/L for kaolin. Therefore, the effect of Fe content in mineral on the removal ratio of As(III) is lower than that of As(III) concentration under existing conditions in this study. This is because hematite loaded can reduce the surface negative charge of kaolin, leading to the decrease of the electrostatic repulsion. Furthermore, despite the reaction systems with As(III) concentration of 24 mg/L, the maximum adsorption amounts of As(III) on minerals are not obtained.

At similar analysis, at a As(III) concentration of 6 mg/L, the removal amount of As(III) adds 2.45 mg/L with Fe content increasing (or kaolin concentration decreasing) from kaolin to He@kaolin-1 at pH 4, while that with an addition of 0.62 mg/L at As(III) concentration from pH 4 to pH 8 for kaolin. Therefore, the effect of Fe content in mineral on

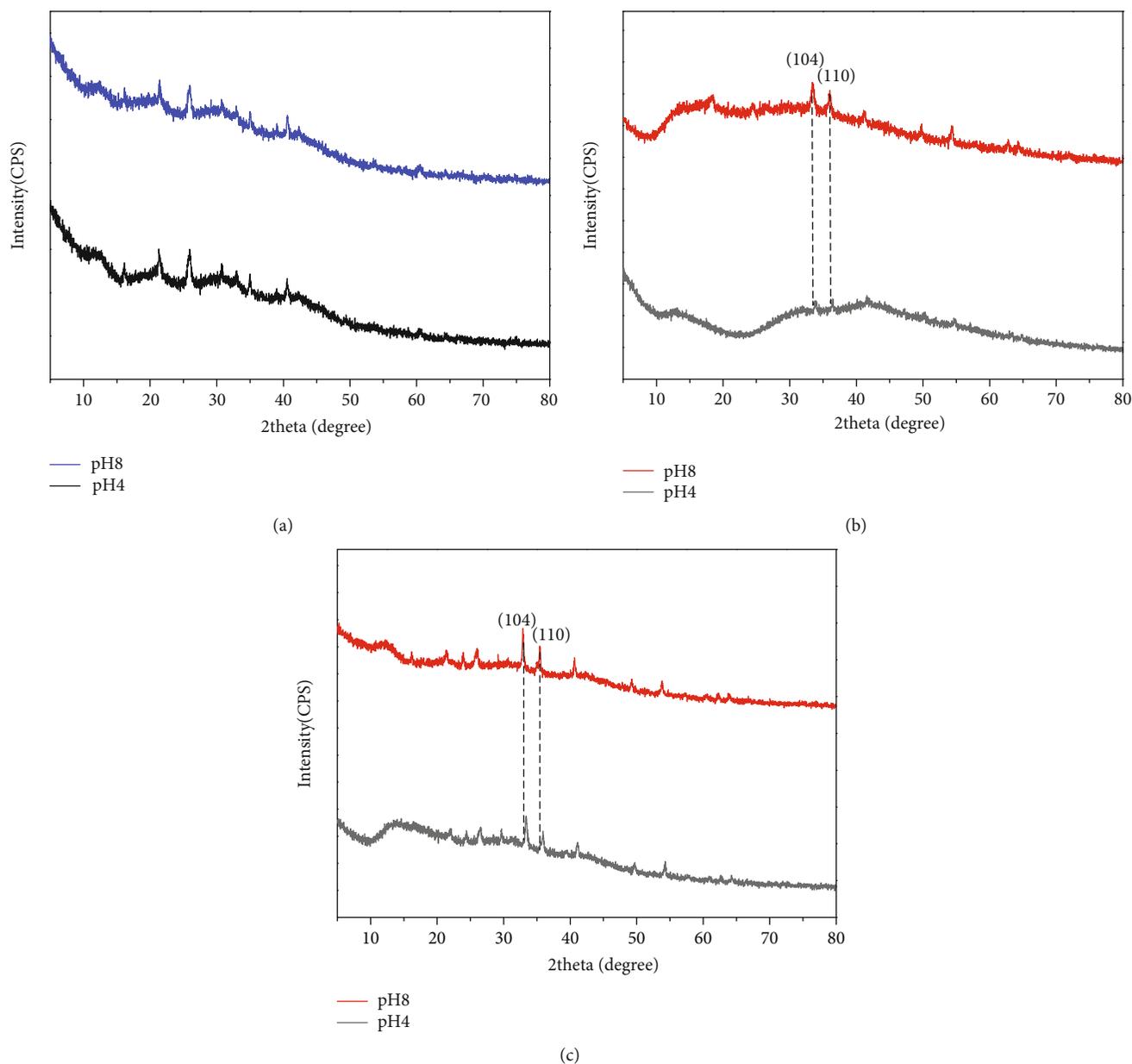


FIGURE 8: XRD patterns of the adsorption products of kaolin (a), He@kaolin-3 (b), and He@kaolin-1 (c) at pH 4 and 8 with 6 mg/L As(III).

the removal ratio of As(III) is greater than that of pH. It indicates that hematite loaded have important influence on the removal ratio of As(III) of kaolin. Furthermore, the adsorptions of kaolin and complexes for ions with negative electric charge are usually gradually decreased with increasing pH [27–29], but the results of the removal ratio of As(III) is contrary. Because the  $PZC_{pH}$  of the minerals can be within the pH range from 4 to 8, the adsorption of As(III) species is favoured electrostatically up to the  $PZC_{pH}$  of the adsorbents; but beyond this point, specific adsorption (ligand exchange) plays an important role [24, 29, 38, 39].

**4.2. The Effect of Proton and Arsenic Concentration on As(III) Removal.** Based on all above results, the adsorption of kaolin for As(III) is relatively low than that of modified

kaolin. And through analysis of the adsorption amount of As(III) at different pH and concentrations, hematite loading can significantly improve the adsorption of kaolin. Furthermore, through normalization analysis adsorption of kaolin and He@kaolin-1: at As(III) concentration of 6 mg/L and 24 mg/L, increasing unit concentration can add 0.21 and 0.32 mg/L As(III) for kaolin and He@kaolin-1; and at pH 4 and 8, increasing unit pH can add 0.16 and 0.19 mg/L As(III) for kaolin and He@kaolin-1. Therefore, hematite loading is advantageous to adsorption of As(III) with increasing As(III) concentration, but that adds less adsorption of As(III) with increasing pH. The results indicate that As(III) concentration has more influence on adsorption of As(III) than pH for kaolin and He@kaolin-1, especially for He@kaolin-1.

## 5. Conclusion

Kaolin clay has a good activity for contaminants in the environment. And a large amount of hematite usually is attached to other minerals' surface leading to increased adsorption ability in the tropical and subtropical regions. Therefore, the complex of hematite loaded on kaolin was synthesized; then, the removal behavior for As(III) is further study. The results show that hematite loading impacted the structure order of part minerals in kaolin and increased the activity sites. In addition, the increase of As(III) concentration and pH can increase the removal amount of As(III) by kaolin, He@kaolin-3, and He@kaolin-1, especially for the concentration of As(III), in which increasing Fe content can also further promote the removal amount of As(III). At last, no forming new mineral was observed in the adsorption products. All above results indicate that the migration and effective concentration of arsenic pollutants can be controlled through increasing pH and/or iron oxide, which can provide reference for remediation of arsenic pollution in the soil or ground water.

## Data Availability

All the basic data that support the results of this study are contained in the article.

## Conflicts of Interest

The authors declare that they have no conflicts of interest.

## Acknowledgments

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