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REMOVAL OF ARSENIC AND HEAVY METALS FROM ARSENIC-CONTAINING ACID WASTEWATER WITH IRON SALT AND LIME

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Abstract

Arsenic-containing acid wastewater produced in the mining and smelting process will bring great harm to the environment, while to treat arsenic-containing wastewater with iron salt and lime is an economical and effective method. In this paper, iron salt was added to co-precipitate the arsenic and heavy metals, the pH value of arsenic containing acid leaching solution was adjusted by adding of lime. The stability of co-precipitates was also investigated in this study. The results show that the iron, arsenic, copper and zinc in the leaching solution can be removed to less than 0.6 mg/L. In addition, arsenic removal is greatly affected by temperature and the molar ratio of Fe/As. The leaching toxicity test of the co-precipitates presents that the precipitates can be stably stored. SEM and XRD analyses of the co-precipitates indicate that the particles size of the co-precipitates is only several microns, and the ferric arsenate still appears in the amorphous state. Moreover, there are crystal calcium arsenate and calcium sulphate which can improve the stability of the co-precipitates.

Key words: arsenic, co-precipitation, ferric arsenate, leaching toxicity, wastewater

Received: March, 2019; *Revised final:* April, 2019; *Accepted:* June, 2019; *Published in final edited form:* December, 2019

1. Introduction

Arsenic-containing wastewater produced in the process of mining and metallurgy is an important source of arsenic pollution. In view of the great potential danger to human health and environment, safe disposal of arsenic wastewater has received widespread attention all over the world (Choong et al., 2007; Jang et al., 2017; Ungureanu et al., 2018).

Among all the methods, lime precipitation is widely used because of its simple process and easy implementation. The treatment mechanism is to add lime to arsenic-containing wastewater to form calcium arsenate, calcium arsenite and other precipitates, and most of the arsenic precipitated from the water is removed by filtration (Bothe and Brown, 1999a; 1999b). Calcium arsenate precipitation is not suitable

for long-term stable storage because CO₂ in the air can transform calcium arsenate into calcium carbonate and releases arsenic from precipitates to pollute the environment again (Robins, 1981; Liu and Zhu, 2006). In contrast, ferric arsenates are more stable than the calcium arsenates owing to their lower solubility. Due to the influence of reaction conditions, the solubility of co-precipitates arsenic with iron varies greatly, among which scorodite is considered the most stable compound (Majzlan et al., 2012; Paktunc and Bruggeman, 2010). However, the synthesis of scorodite usually requires high temperature and high pressure, and the harsh reaction conditions greatly reduce the value of its industrial application. Amorphous ferric arsenate is generally formed at room temperature and atmospheric pressure (Taboada et al., 2009; Wang et al., 2018a), and its formation is

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also accompanied by the adsorption of arsenic onto ferric hydroxide (Müller et al., 2010). As a result, ferric arsenate obtained in most cases has poor stability and can easily release arsenic into water (Krause and Ettl, 1989).

However, it is reported that the stability of ferric arsenate obtained under the condition of the molar ratio of Fe/As greater than three can be greatly improved, and it is generally considered that it can be stored safely for a long time (Singhania et al., 2006; Krause and Ettl, 1988). The process of arsenic precipitation with iron oxides is greatly affected by the pH value of the solution. In laboratory research, NaOH is usually employed to adjust the pH value. However, lime is normally used to adjust the pH due to its economy in the industrial process (Cui et al., 2014; Klerk et al., 2014; 2015). When lime is used instead of the NaOH to adjust pH value, not only the stability of co-precipitates can be improved (Jia and Demopoulos, 2008), but also the calcium ion at higher pH value can enhance the ability of Fe(OH)₃ to adsorb arsenic (Liu et al., 2007).

In addition to ferric arsenate, there is also a large amount of gypsum in the co-precipitates (Riveros et al., 2001). It has also been argued that the presence of gypsum can improve the long-term stability of co-precipitates (Harris and Krause, 1993). In some industrial production, the wastewater from the actual mining and metallurgical process contains a large amount of arsenic and some heavy metals, such as copper, zinc, nickel, or cobalt. These divalent ions may also co-precipitate with the arsenic and iron.

There is evidence that these ions have little effect on the scorodite stability (Fujita et al., 2008; Singhania et al., 2006). However, these ions will also bring a significant risk to the environment if they remain in the wastewater. Therefore, the leaching solution containing arsenic, iron, copper and zinc ions was chosen as the research object to study the co-precipitation behaviour in the solution, and the stability of the co-precipitates.

2. Experimental

2.1. Materials and analytical methods

The arsenic containing leaching solution was obtained through the removal of arsenic from arsenic containing pyrite cinder in the laboratory. The leaching test was carried out using sulfuric acid at normal temperature and atmospheric pressure. And the leachate was collected and homogenized after the reaction.

The concentrations of As, Fe, Zn, and Cu in the solution were determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Optima 8000) (Detection limit of As, Fe, Zn, and Cu are 0.03, 0.002, 0.005 and 0.005 mg/L, respectively). The co-precipitates were characterized by a scanning electron microscope (SEM, JSM-7001F+INCA-MAX), an X-ray diffraction (XRD, X'Pert PRO

MPD). The pH value of the solution was examined by a pH tester meter (Mettler Toledo FE20).

The leachate was tested by the pH tester meter, and the results showed that the pH value was about 0.5. The ICP results show that the concentrations of arsenic, ferric, copper and zinc in the leachate were 8011.2 mg/L, 5995.1 mg/L, 278.8 mg/L, 712.8 mg/L, respectively. The leachate contains a large amount of iron and arsenic, and the molar ratio of Fe/As is calculated to be about 1:1, which is conducive to the co-precipitation of arsenic with iron.

2.2. Experimental methods

(1) Co-precipitation

As(III) is much more toxic and less stable than As(V), thus a given volume of hydrogen peroxide was added to the leaching solution at first and stirred continuously for six hours, which can make As(III) fully oxidize to As(V) (Feng et al., 2018; Wang et al., 2018b; Stephan and Olivier, 2003). Meanwhile, the leaching solution also contains lots of Fe(II) ions, which will be oxidized to Fe(III). A certain volume of pre-oxidized leaching solution was added to a beaker flask, and ferric sulphate was added reach the desired molar Fe/As ratio. After stirring evenly, lime powder was added to the solution to adjust the pH value. When the pH reached the desired value, the precipitates were separated from the solution rapidly by vacuum filtration. The concentrations of Fe, As, Cu and Zn in the filtrate were determined by ICP-OES, and the leaching residue was detected by SEM-EDS and XRD.

(2) Leaching toxicity test

In this study, the acetate buffering solution of pH=4.95±0.05 was used in the leaching toxicity test. The co-precipitates were put into a centrifugal tube, and the acetate buffer solution was dripped to reach the solid to liquid ratio of 1:20. The tube was sealed, fixed in an oscillator with vibrating frequency at 110 times/min, and shaken 40 hours at room temperature. Then, the leaching solution was filtered using a vacuum filter to separate the solid and liquid. The separated liquid was used to analyse the arsenic concentration by ICP.

3. Results and discussion

3.1. Effect of pH value

The lime powder was used to adjust the pH value of the leaching solution in this study. The concentrations of Fe, As, Cu and Zn obtained at different pH values are given in Fig. 1. The results show that concentrations of all four elements decrease with the increasing pH value. The As concentration decreases rapidly in the pH range of 2-4. It decreases down to 9.1 mg/L at pH = 4.12 and shows no noticeable change upon further pH increase. When the pH value exceeds 9, arsenic concentration fluctuates in the range of 2-3 mg/L.

The Fe concentration in the solution decreases rapidly and drops to 11.99 mg/L at pH=2.36. Iron could not be detected in the solution at pH above 4.12 (detection limit of Fe is 0.002 mg/L). The Cu concentration in the solution decreased almost linearly in the pH range of 2-5. The copper concentration is 7.09 mg/L at pH = 4.95. On further increase to pH=5.63, the Cu concentration is <0.3mg/L. The Zn concentration decreased slowly in the pH range of 2-5, but the concentration decreased rapidly when the pH is greater than 5. The zinc concentration is 4.89 mg/L at pH=5.63. When pH increases to more than 7, it reduces to <0.3 mg/L.

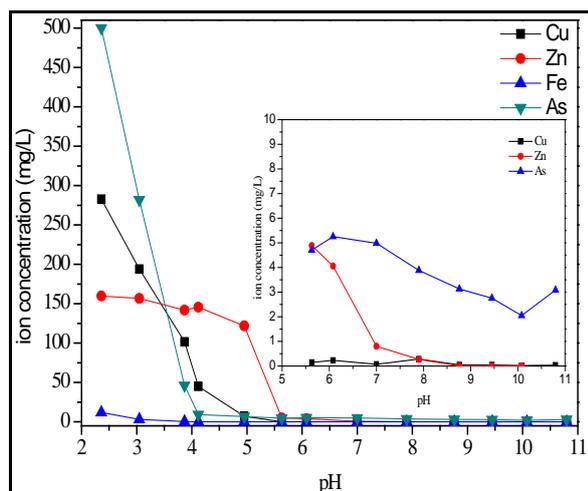


Fig. 1. Concentrations of arsenic, iron, copper and zinc under different pH values

In summary, the concentrations of Fe, Cu and Zn in the solution will gradually decrease to below 0.5 mg/L with the increase in pH value. The lowest concentration of arsenic is between 2-3 mg/L and is difficult to be suppressed below 0.5 mg/L. Meanwhile, calcium arsenate can be also produced using the lime to adjust the pH value.

Both of the calcium arsenate and iron arsenate with amorphous state have a high solubility in water, which leads to a higher solubility of arsenic in the solution. Comparing the results with our previous study, the arsenic concentration decreased to 8.19 mg/L when the ammonia was used to adjust the pH value under the condition of Fe/As = 1:1 (Wang et al., 2018a). This also proves that the presence of calcium will improve the arsenic removal effect (Jia and Demopoulos, 2008; Liu et al., 2007).

3.2. Effect of Fe/As molar ratio

A certain amount of ferric sulphate was added to the leaching solution to adjust the Fe/As molar ratio. After dissolution and stirring, the lime powder was added to adjust the pH value at 5.5 ± 0.2 , and the results of different ion concentrations under different Fe/As are presented in Fig. 2.

The results show that the increase of Fe/As has a negligible effect on the removal of Fe, Cu and Zn.

The concentration of zinc fluctuates in the range of 8-12 mg/L, and the concentrations of iron and copper are below 0.2 mg/l and 0.6 mg/L, respectively. However, the As concentration decreases below 0.6 mg/L when the Fe/As ratio is between two and four. Therefore, the removal of arsenic from solution benefits from the increase of the Fe/As ratio.

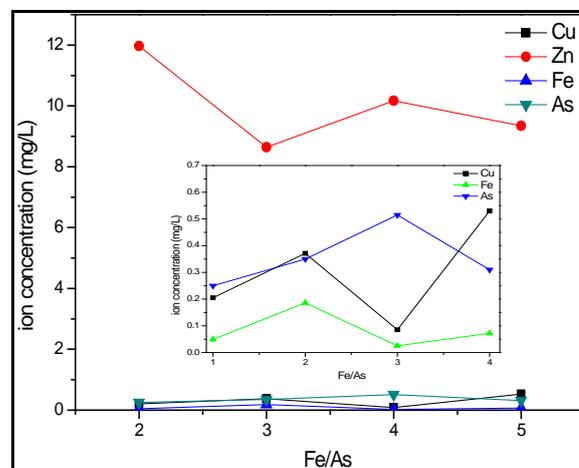


Fig. 2. Concentrations of arsenic, iron, copper and zinc under different Fe/As molar ratios

3.3. Effect of temperature

The leaching solution was heated to a desired temperature in a water bath, and the lime was added to adjust the pH to 5.4 ± 0.3 . The results of different ion concentrations between 40-80°C are shown in Fig. 3. The temperature has little effect on the removal of Fe, Cu and Zn, and their concentrations fluctuate in a narrow range. The concentrations of Fe and Cu are both less than 0.5 mg/L, and the concentration of zinc is in the range of 11-18 mg/L. However, the concentration of arsenic increases with the increasing temperature.

The concentration of arsenic is only 8.4mg/L at the temperature of 40 °C . When the temperature reaches to 80°C , the As concentration reaches 23.4 mg/L. It can be speculated that the co-precipitates produced under these conditions contain a large amount of poorly crystalline ferric arsenate and calcium arsenate, whose solubility increases with the temperature increasing. This may be the reason of the arsenic concentration increasing with the temperature (Wang et al., 2018a).

3.4. Leaching toxicity of co-precipitates

The leaching toxicity test of the co-precipitates obtained at different pH values was conducted at room temperature. After the test, the pulp was separated into solid and liquid, and the filtrate was tested by ICP-AES to determine the concentrations of Fe, Cu, Zn and As. The results show that these elements cannot be detected in the filtrate. The pH value of the acetate buffering solution is only 4.95 ± 0.05 , so the ferric iron will not be leached under this condition. The contents of copper and zinc contained in the co-precipitates are

quite low. The copper content is only 0.08-0.2%, and zinc content is 0.2-0.5%. Moreover, copper and zinc may produce stable compounds in the co-precipitates. Therefore, the leaching toxicity is very low, and cannot be detected. The leaching toxicity results of arsenic are presented in Fig. 4. As it was shown in this figure, the leaching toxicity of arsenic fluctuates within the range of 0.2-0.9 mg/L. The effect of pH value on the leaching toxicity is not obvious. Compared with our previous study (Wang et al., 2018a), when ammonia was utilized to regulate the pH value, the arsenic leaching toxicity of the co-precipitates is 39.41 mg/L at Fe/As = 1:1.

Therefore, the use of lime is beneficial for the stability of co-precipitates. The leaching toxicity of co-precipitates obtained from solutions with different Fe/As molar ratios was also studied. The arsenic leaching toxicity of the precipitates is further diminished as the Fe/As molar ratio increasing. When the ratio increases to 2:1, the arsenic leaching toxicity decreased to less than 0.2 mg/L.

However, the leaching toxicity is not obvious change when the Fe/As ratio is further increased. The results show that when the lime was used to adjust the pH value, the leaching toxicity of the co-precipitates was greatly affected by the production of the calcium arsenate.

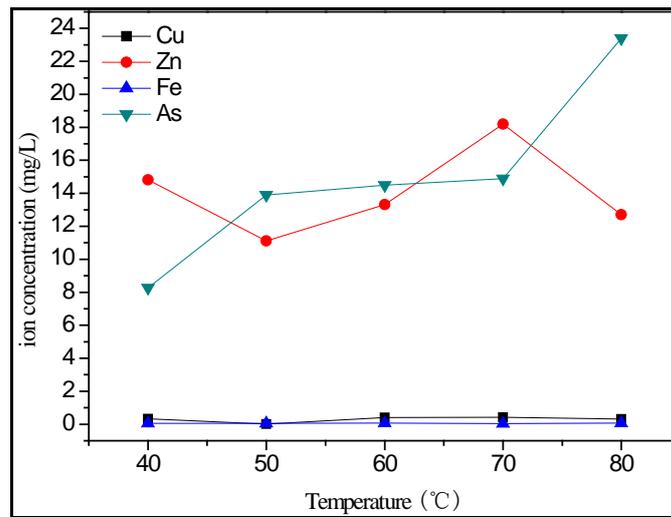


Fig. 3. Concentrations of arsenic, iron, copper and zinc under different temperatures

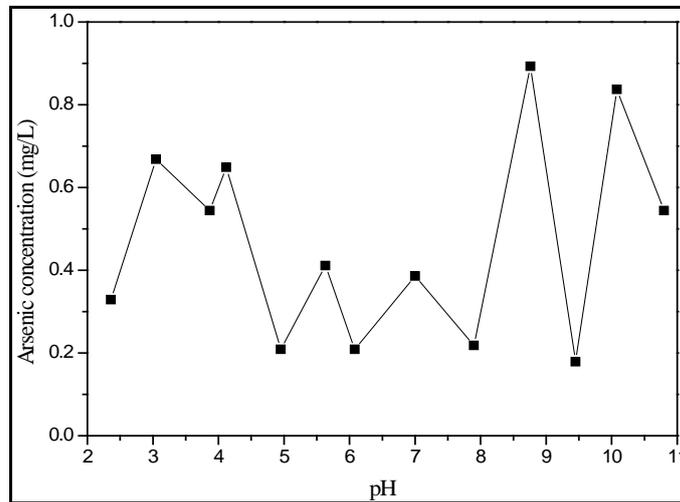


Fig. 4. Arsenic leaching toxicity of co-precipitates obtained under different pH values

However, the effect of lime on the leaching toxicity is not obvious as Fe/As > 2, which may be due to the high solubility of calcium arsenate.

3.5. Discussion

The co-precipitates obtained under different pH values were analyzed by XRD and SEM, and the

results are shown in Fig. 5 and Fig. 6. The XRD results show that the composition of the precipitates obtained under lower pH conditions is relatively simple. At pH=2.36, the main phase is CaSO₄·0.5H₂O (Bassanite), and the peaks of CaSO₄·2H₂O (Gypsum) and CaH(AsO₄)·2H₂O (Pharmacolite) also appear. The acid leaching solution is mainly obtained from the removal of arsenic from pyrite cinder using sulfuric

acid, so there is a large amount of sulfate radical in the solution.

When lime is added, the gypsum will inevitably form because of its low solubility, and the main reactions are as follows Eqs. (1-2):

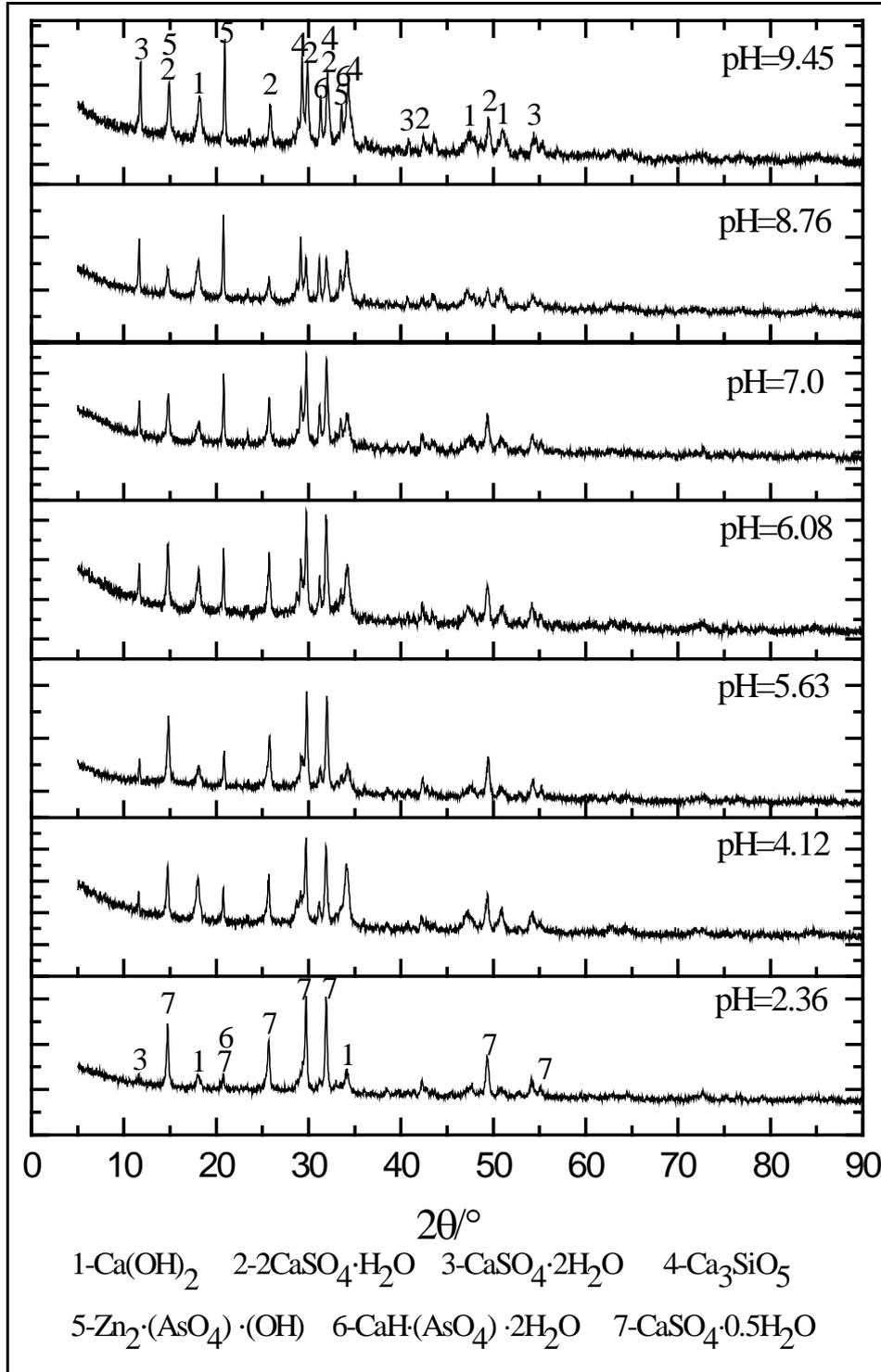
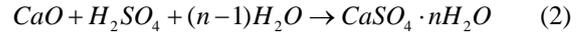
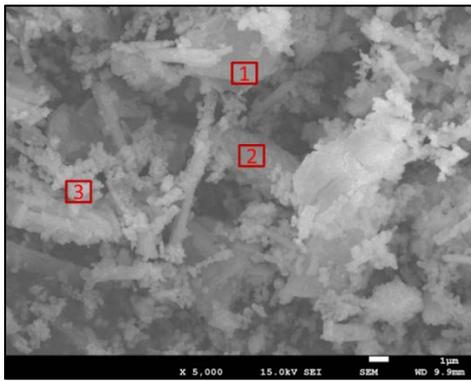
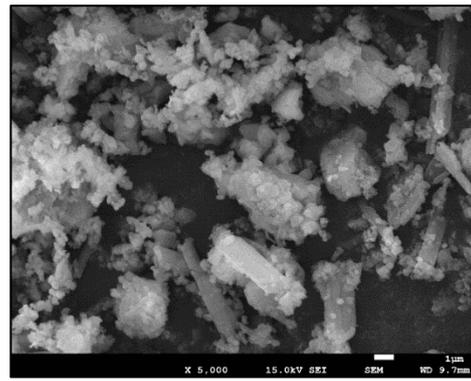


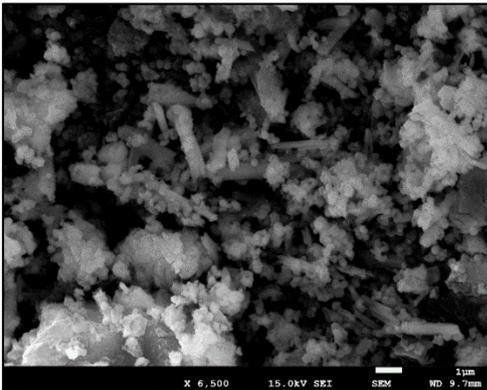
Fig. 5. XRD results of the co-precipitates obtained under different pH conditions



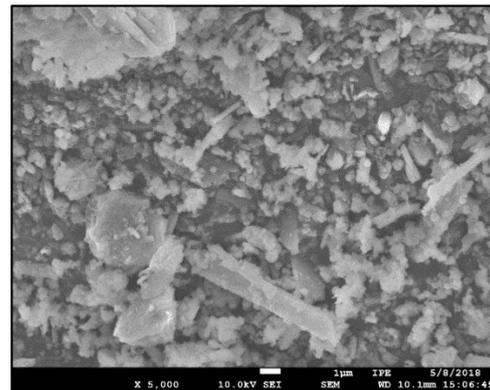
(a)



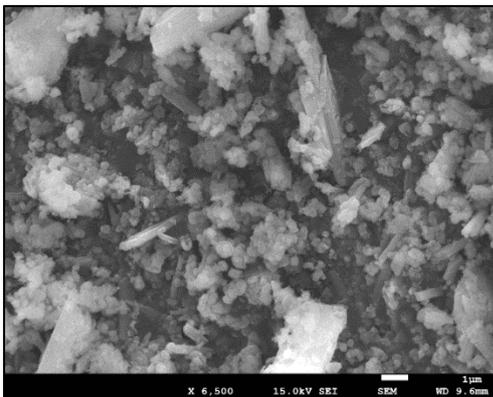
(b)



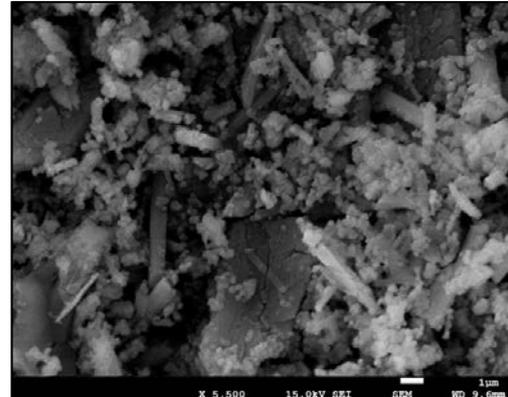
(c)



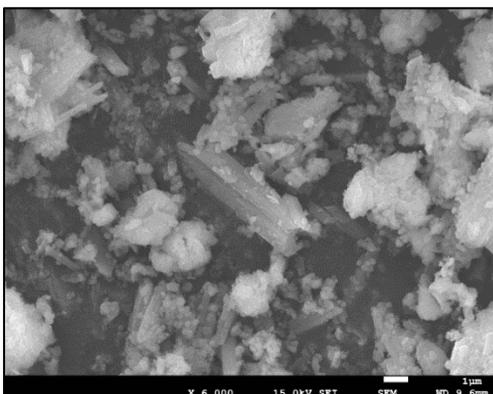
(d)



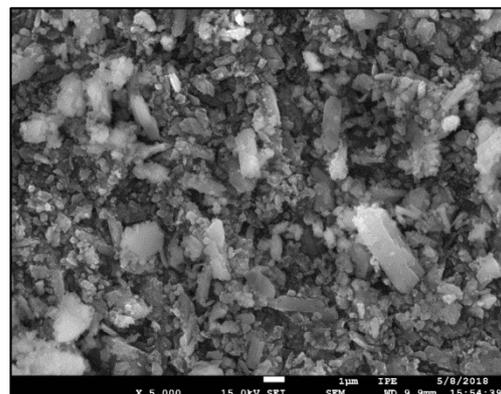
(e)



(f)



(g)



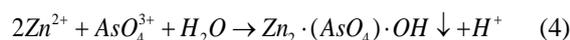
(h)

Fig.6. SEM images of the co-precipitates obtained under different pH conditions: (a) pH=2.36; (b) pH=4.12; (c) pH=4.95; (d) pH=5.63; (e) pH=6.07; (f)pH=7.0; (g) pH=8.76; (h)pH=9.45

Calcium ions will combine with arsenate to produce crystalline precipitates (see Eq. (3)), which is also conducive to the stable storage of arsenic.



The intensity of the XRD peaks gradually increases with increasing pH, and more peaks began to appear at pH=4.12. At pH =9.45, the XRD peaks appeared more intensively, especially in the peak range of 25-35°. In addition to the calcium sulfate hydrate, there are also the peaks of $CaH \cdot (AsO_4) \cdot 2H_2O$, $Zn_2 \cdot (AsO_4) \cdot OH$ (Adamite) and Ca_3SiO_5 (Calcium Silicate Oxide). The Eqs. (4-5) may occur:



However, the XRD peak of ferric arsenate in the pH range of 2.36-9.45 could not be found, which means that ferric arsenate in the precipitates is still X-ray amorphous. This indicates that the reduction of leaching toxicity of co-precipitates is not due to the increase of the crystallinity of ferric arsenate, but is more closely related to the crystals of $CaH \cdot (AsO_4) \cdot 2H_2O$ (Camacho et al., 2009).

Fig. 6 presents the SEM results of co-precipitates obtained under different pH conditions. The SEM images demonstrate that the particle size of the co-precipitates is only a few microns. The particle size has little change in different images, and this indicates that the pH changes have usually little effect on the particle size.

Rod-shaped particles appear in the co-precipitates and it can be inferred from the results of energy-dispersive analysis (Table 1) that the main component is $CaSO_4$. Previous studies showed that the presence of calcium sulfate can also improve the stability of arsenic in co-precipitates (Harris and Krause, 1993). In addition, the results in Table 1 show that the size of the particles formed by arsenic co-precipitated with iron is smaller, as it is shown in Fig. 6(a) where the arsenic and iron occur together at the point 3. There are no XRD peaks in Fig. 5 that could be assigned to crystalline ferric arsenate, meaning that the ferric arsenate mainly exists in the X-ray amorphous form.

Little part of the arsenic is distributed on the surface of the rod as shown at point 2 in Fig. 6(a), which may produce calcium arsenate. The above results show that when lime is used to adjust the pH value, both of the crystallinity of ferric arsenate and the particle size of the crystal will not be increased.

4. Conclusions

The main parameters that influence removal of As, Fe, Cu and Zn in the treatment of arsenic-containing wastewater with iron salt and lime are pH, Fe/As ratio and temperature. When pH is greater than

5.5, the concentrations of Fe, Cu and Zn in the solution drop to <0.5 mg/L.

Table 1. SEM/EDS results of different points in Fig. 6(a)

Element	1		2		3	
	Atomic/%	Weight/%	Atomic/%	Weight/%	Atomic/%	Weight/%
O	78.11	61.31	78.59	61.2	75.43	51.22
S	11.04	17.36	9.96	15.55	5.33	7.26
Ca	10.85	21.33	10.91	21.28	11.62	19.77
Fe	-	-	-	-	3.04	7.21
As	-	-	0.54	1.97	4.57	14.53
Totals	100	100	100	100	100	100

However, the arsenic concentration decreases just to about 9.1 mg/L. Increasing the Fe/As molar ratio is beneficial to the arsenic removal and can diminish the aqueous concentrations to <0.6 mg/L at Fe/As = 2-4.

At temperatures below 80°C, increasing temperature is not conducive to the removal of As, and has little effect on the removal of Fe, Cu and Zn. Leaching toxicity results show that the stability of co-precipitates with lime as neutralizer is better when the ratio of Fe/As=1, and the leaching toxicity is less than 1 mg/L.

Increasing the Fe/As ratio can improve the stability of co-precipitates. The appearance of $CaH \cdot (AsO_4) \cdot 2H_2O$ and $CaSO_4$ during the lime neutralization process may be a major reason for the stability improvement in the co-precipitates. The ferric arsenate is mainly X-ray amorphous, using lime as the neutralizer does not increase the crystallinity of ferric arsenate.

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