Removal of arsenic from zinc oxide leaching solution by oxygen combined with ultrasonic

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Research Article

Keywords: Ultrasound, redox, chemical precipitation, arsenic removal

Posted Date: September 9th, 2022

DOI: https://doi.org/10.21203/rs.3.rs-2041158/v1

Abstract

In this study, the oxidation method combined with the iron salt precipitation method was used to remove the arsenic impurities in the zinc oxide fume leaching solution after germanium precipitation. First, the Fe2+ and As5+ in the solution are oxidized by oxygen with a specified flow rate into the solution, and then the calcination fume is added to adjust the pH value of the solution with Zn compounds such as ZnO, so that the impurity elements in the solution are precipitated. The effects of reaction time, reaction temperature, roasting soot input, oxygen flow rate and ultrasonic power on the arsenic removal rate were investigated in the experiment. The experimental results show that under the conditions of reaction time of 120min, reaction temperature of 70℃, roasting soot input of 7g, ultrasonic power of 240W and oxygen flow of 0.8L/min, the removal rate of arsenic can reach 92.77% by two-stage arsenic removal. , the loss rate of zinc is only 2.54%, and the amount of dry slag after removal is 5-7g. Compared with the iron salt precipitation method in the traditional industry, it has the advantages of less slag, less zinc loss, no harmful by-products, etc., and the main raw materials used oxygen and roasting dust are cheap products, which can save costs. The arsenic content in the finally obtained filtrate can reach 0.125g/L, and less than 0.2g/L can meet the requirements of the subsequent electrolysis process, and can be used for practical industrial production applications.

Environmental Implications

This research helps to solve the impurity problem in the industrial process, can effectively remove arsenic in the zinc oxide leaching solution after germanium precipitation, and make the subsequent electrolysis process more energy-efficient and efficient.

1. Introduction

Preliminary research analysis, currently the main fire-fighting engineer in our country, during this process, the temperature rises and increases due to the temperature increase [1-3], the formation part of the smoke Collected ash formed after cooling the gas flow, various useful elements contained in the ash [5-7], such as gold, brass, etc. Therefore, the world&apos,s ability to reduce the number of products is currently being developed, and various methods have been developed to supply the available elements. It is possible to use different types and contents of different elements [8-9], and it is possible to use different methods, among which the wet leaching method is most widely used. Xin [4] adopted Ultrasonic-H_2O_2 (UH) Combined Moisture-Leached Powder (ZOD) Medium-selective Zn Sum Ge. Leaching temperature (50~90°C) and leaching time (30~240 min) Zn and Ge specific leaching rate progress research. The results were announced, the best leaching conditions were: ultrasound power rate 200 W, H2O2 dosage 14.8 mL, primary acidity 160 g L−1, liquid-solid ratio 7 : 1, leaching time 60 min, stirring speed 400 rpm, leaching temperature 60 ℃, kettle. Japanese leaching rate is 99.61% and 88.29%.

Arsenic is a protoplasmic toxic element with metalloid properties and has a wide range of biological effects. It has been identified as a Group 1 carcinogen by the US Centers for Disease Control and the

International Institute for Cancer Prevention. If there are too many arsenic ions in the solution, it will form an extremely harmful acidic solution - foul acid [10-12]. At present, the commonly used arsenic removal methods can be summarized as coagulation method, precipitation method and ion exchange method, adsorption method [13-15], reverse osmosis method, biological method and so on. The coagulation method mainly uses the strong adsorption effect of the coagulant (large active surface area) to adsorb arsenic, and then filters or uses a filter membrane to remove the arsenic in the water [16-18]. The method is currently the most widely used arsenic removal method in industrial production and drinking water treatment, which can well make industrial sewage meet the discharge standard and the drinking water to meet the drinking standard. The precipitation method mainly utilizes external agents or energy to have chemical or physical interaction with arsenic pollutants in water to form precipitates or flocs [19-21], and then separate the precipitate from the aqueous solution to achieve the purpose of arsenic removal. Adsorption method is a simple and easy wastewater treatment technology. It is generally suitable for water treatment systems with large processing capacity and low concentration. Its advantage is that it can remove harmful substances in wastewater without increasing the salinity of the water body [22-24]. It is a common method for secondary treatment of arsenic-containing wastewater. The disadvantage is that there is a strong adsorption effect between the adsorbent and the arsenic compound, which often makes the adsorbent unable to regenerate, and it is difficult to recycle and reuse [25-26]. Oxidative arsenic removal is an effective method to remove trivalent arsenic, but the reaction kinetics are often slow and the investment is large, which limits its application to a certain extent. The ion exchange method can only treat wastewater with low concentration, small treatment volume, simple composition and high recovery value [27-29]. The advantage of biological arsenic removal is that it can purify water, but the disadvantages are also obvious. The required raw materials and related technologies are very expensive and are not suitable for large-scale arsenic removal treatment.

The zinc oxide flue gas leaching solution used in this paper is the solution obtained by leaching the zinc element from the above-mentioned soot by sulfuric acid and then by germanium precipitation. By this method, most of the zinc in the dust can be leached into the sulfuric acid solution, and the effect of germanium precipitation is also very prominent, which can achieve a good separation effect of valuable elements. However, due to the inability to achieve directional leaching in the process, some harmful impurities will also be leached into the solution, the most serious of which is the toxic element-arsenic. The experimental results show that even if the leaching rate of arsenic in the solution is low, the arsenic content in the leaching solution still reaches 1.73 g/L, which is enough to affect the subsequent zinc sulfate electrolysis process, so we must take further measures. Arsenic is removed from this solution to eliminate its effect on subsequent processes. Through elemental analysis of the solution used, it was found that the solution itself contained a part of iron, and the valence states of arsenic and iron in the solution were both low-valence states. Therefore, it was finally decided to use the oxidation method combined with the iron salt precipitation method to remove arsenic. One of the main innovations of this study is to use oxygen as an oxidant to oxidize Fe^{2+} and As^{3+} in the leaching solution. The ideal oxidation effect can be achieved by ultrasonic synergy. The roasting fume is used as a neutralizing agent, and Zn compounds such as ZnO can be used as neutralizers to a good effect of adjusting pH. The advantage of the above method is that the raw materials used are cheap and easy to prepare, and the defect of low oxygen oxidation efficiency can be well overcome by ultrasonic synergy. The whole experimental process does not produce any harmful by-products, which can achieve the effect of energy saving and emission reduction.

2. Experiment And Methods

2.1 Experimental raw materials

The raw material used in this experiment is a solution obtained by immersing the dust collected during the pyrometallurgical smelting of zinc-gold ore in concentrated sulfuric acid, leaching the valuable elements of zinc in it, and then subjecting the resulting leaching solution to germanium precipitation. The initial pH of the solution is approximately 1.4. The main elements and their contents in the solution are shown in Table 1.

Table 1 chemical composition analysis of leaching solution

Component	As	Fe	Cd	Cu	Mg	Zn
Content(g/L)	1.73	1.89	0.349	0.0003	28.26	109.72

It can be seen from the above table that the main element in the leaching solution is zinc, and the main impurity element to be removed is arsenic, which also contains a certain amount of iron.

The neutralizer roasting dust used in this experiment comes from the dust obtained from the fire roasting of zinc sulfide concentrate. Its main components are related compounds of zinc. The analysis of the XRD results in Figure 1 shows that the main phases of zinc in the dust are ZnO, ZnS, $ZnSO_4$. The advantage of using this neutralizer is that it can also adjust the pH of the solution without introducing a large amount of other elements into the solutionl

The following are other materials and related equipment required for the experiment:Cp114 electronic balance, circulating water multi-purpose vacuum pump, lzb-3wbf glass rotameter, yqy-12 oxygen reducer, intelligent ultrasonic generator, df-101s heat collecting constant temperature heating magnetic stirrer, burette, three port flat bottom flask, 500ml measuring cylinder, beaker, pH test paper.

2.2 Experiment principle

According to the characteristics of As and Fe in the leaching solution and their valence analysis in the solution, the chemical precipitation method combined with the oxidation method was used to remove arsenic in this experiment [31-32]. Combined with the relevant ionic thermodynamic data given in the data, the Eh-pH diagram of the As-Fe-H_2O system was drawn (Fig. 2). For the convenience of calculation and drawing, it is assumed that the activity of each ion in the solution is 1.0, and that PH_2 and $PAsH_3$ are 1.0 atm. As can be seen from the figure, when the pH value of the solution is 1.4, the ions present in the solution are As^{3+} and Fe^{2+}. In order to make them meet the requirements of forming precipitates [33-34], they must be oxidized first. In this experiment, oxygen was used as an oxidant combined with ultrasonic waves for oxidation. Possible reactions during this process are:

$2H_3ASO_3+O_2===2H_3ASO_4$

$2Fe^{2+}+O_2===2Fe^{3+}+O^{2-}$

After complete oxidation by O_2 combined with ultrasonic waves, most of As^{3+} and Fe^{2+} in the leaching solution have been converted into high valence As^{5+} and Fe^{3+}. When the pH value of the solution exceeds 1 [35-36], Fe^{3+} in the solution will combine with H_3AsO_4 and convert to $FeAsO_4$. When the pH value of the solution continues to rise to 5, $FeAsO_4$ can be converted into $Fe(OH)_3$ and $H2AsO_4^-$, and then $H2AsO_4^-$ will be adsorbed by $Fe(OH)_3$ and precipitated together with iron. The main reactions involved in this process are:

$Fe^{3+}+H_3AsO_4===FeAsO_4+3H^+$

$FeAsO_4+2H_2O+OH^-===Fe(OH)_3+H_2AsO_4^-$

2.3 Experimental method

Measure 500ml of zinc oxide leaching solution after germanium deposition, put it into a three-necked flat-bottomed flask, put the flask into a water bath, turn on the stirrer, and begin to heat up gradually. After the temperature rises to the specified temperature, a certain flow of oxygen is introduced, and the neutralizing agent is gradually added to adjust the pH value of the solution to 4.0-4.5. After the solution has reacted for a certain period of time, use a vacuum pump to separate the slag liquid, then pour the filtered solution back into the flask, continue to introduce oxygen and stir, add a neutralizer to adjust the pH to 5.0-5.4, and wait for the solution to react at the same time as the previous period. After that, vacuum filtration is performed again. Finally, the dry slag obtained from the two experiments was dried and weighed, and the XRD phase analysis was carried out. The arsenic content in the filtrate was obtained by elemental analysis, and the arsenic removal rate was calculated.

3. Results And Discussion

3.1 Oxidation of Arsenic and Iron

According to the thermodynamic analysis of the zinc oxide leaching solution used in this experiment after germanium is deposited, it can be seen that the arsenic in the solution mainly exists in the form of $HAsO_2$. Trivalent arsenic is highly toxic, and it is not easy to produce precipitation to remove, so it needs to be oxidized first. In this experiment, the excellent and obvious oxidizing property of oxygen is used, and oxygen bubbles are used as the oxidant. At the same time, the synergistic oxidation by the cavitation of ultrasonic waves can well overcome the disadvantage of low oxygen oxidation efficiency. The results show that the oxidation efficiency of oxygen can be greatly improved under the cooperation of ultrasonic waves. The oxygen flow and ultrasonic power introduced during the experiment are the two main factors affecting the final arsenic removal rate. On the other hand, according to the characteristics of the oxidant oxygen used in this experiment, its solubility in the solution decreases with the increase of temperature, which also directly affects the final oxidation degree of arsenic. For arsenic removal by chemical precipitation, the arsenic removal rate is proportional to the experimental temperature. Therefore, reasonable control of the reaction temperature of the experiment is also important for the overall arsenic removal rate.

3.1.1 Effects of Ultrasonic Power on Arsenic Removal

Ultrasonic is a sound wave with a frequency higher than 20kHz [37-40], which can produce ultrasonic cavitation effect in the solution, and form local high temperature and high pressure in the solution, which can greatly improve the oxidation efficiency of oxygen. The use of ultrasonic waves will generate local high concentrations of oxide -OH in the solution [41-44], and also has the effect of oxidizing low and low valence ions in the solution. Generally speaking, ultrasonic waves are an excellent auxiliary means for this experiment. In order to verify the actual optimization effect of ultrasonic on this experiment, a comparative experiment with and without ultrasonic-assisted arsenic removal was carried out. The experimental results are shown in Table 2.

Table 2 comparison test results with and without ultrasonic assistance

ultrasonic	Dry slag quantity/g	Arsenic removal rate/%
Yes	5.2264	90.69
No	4.4358	69.54

It can be seen from the above table that ultrasound has a very obvious optimization effect on the final arsenic removal effect of this experiment, which proves that it can indeed make up for the defects of oxygen itself. In order to further strengthen the application of ultrasound, it is necessary to explore the actual test results of ultrasound under different powers.

Take 500ml of the leaching solution, set the temperature of the constant temperature water bath to 55°C, and after the water temperature rises to the specified temperature, turn on the ultrasonic generator, set the power to 180-300W, inject oxygen, and use the oxygen flow rate of 0.6L/min. At the same time, a neutralizer was added to adjust the final pH of the solution to 5.2-5.4, and the total reaction time was 90 min. The effect of ultrasonic power on the arsenic removal effect was investigated, and the results are shown in Figure 4.

It can be seen from Figure 3 that with the continuous enhancement of ultrasonic power, the removal rate of arsenic and the amount of dry slag are also increasing, and the rising slope is the most obvious at

180w-210w and 240w-270w. When the power reaches 270W, the removal rate of arsenic is basically stable, and the continuous enhancement of power has little effect on the improvement of arsenic removal rate. Based on the above analysis, 270W power is more appropriate.

3.1.2 Effects of experimental temperature on arsenic removal

Take 500ml of the leaching solution, set the temperature of the constant temperature water bath to 25-85°C, after the water temperature rises to the specified temperature, turn on the ultrasonic generator, set the power to 240W, introduce oxygen, and the oxygen flow rate is 0.6L/min, add neutralizer to adjust The final pH of the solution was 5.2-5.4, and the total reaction time was 90 min. The effect of experimental temperature on the arsenic removal effect was investigated, and the results are shown in Figure 4.

It can be seen from Figure 4 that before the temperature reaches 40 °C, the removal rate of arsenic changes little with the increase of temperature. When the temperature reaches 55 °C, the removal rate of arsenic increases significantly. When the temperature reaches about 70 °C, the removal rate of arsenic reaches the maximum. If you continue to increase the temperature to 85 °C, the removal rate of arsenic decreases, indicating that the solubility of oxygen in the solution has been low enough to meet the requirements of adequate oxidation of arsenic, At this time, the amount of dry slag obtained from the test is still increasing, which is a phenomenon that the advantages outweigh the disadvantages. Therefore, compared with the traditional iron salt precipitation method, the higher the temperature when using oxygen as oxidant to oxidize arsenic ions and iron ions, the better effect may not be achieved. According to the test results, the best reaction temperature at this time should be about 70 °C, at this temperature, not only the arsenic removal rate is high, but also the amount of dry slag is small.

3.1.3 Effect of oxygen flow on arsenic removal

Take 500ml of leaching solution, set the temperature of constant temperature water bath to 70 °C, after the water temperature rises to the specified temperature, turn on the ultrasonic generator, set its power to 240W and inject oxygen, the oxygen flow rate is 0.2-1.0L/min, add neutralizer to adjust the final pH of the solution to 5.2-5.4, and the total reaction time was 90 min. Investigate the influence of the test oxygen flow on the arsenic removal effect, and the results are shown in Figure 5.

According to the above research on the effect of temperature on arsenic removal, it is found that the effect is the best at 70 °C, and the solubility of oxygen is related to its ultimate oxidation degree of low valence ions in the solution. Therefore, the influence of oxygen flow on arsenic removal effect is explored at this temperature, and the most reasonable oxygen flow is selected. It can be seen from the figure that when oxygen with oxygen flow of 0.2-1.0L/min is introduced, the arsenic removal rate and dry slag volume are constantly increasing. When the oxygen flow is increased from 0.4L/min to 0.6L/min, the arsenic removal rate and dry slag volume increase most significantly. When the oxygen flow continued to increase to 0.8L/min, although the amplitude decreased, there was still a significant increase, and then the flow continued to increase to 1.0L/min without a significant increase. It can be judged that when the temperature is 70 ℃, oxygen with a flow rate of 0.8L/min can play a better oxidation effect, and the arsenic removal rate also reaches a high level.

3.1.4 Effect of reaction time on arsenic removal

Take 500ml of leaching solution, set the temperature of constant temperature water bath to 70 ℃, after the water temperature rises to the specified temperature, turn on the ultrasonic generator, set its power to 240W and inject oxygen, the oxygen flow is 0.8L/min, add neutralize to adjust the final pH of the solution to 5.2-5.4, and the total reaction time was 30-150min. Investigate the influence of the test reaction time on the arsenic removal effect, and the results are shown in Figure 6.

It can be seen from Figure 6 that with the extension of reaction time, the removal rate of arsenic and the amount of dry slag are slowly increasing, and basically reach a stable state after 120min. However, after continuing to extend the reaction time to 150min, the removal rate of arsenic is reduced, and the amount of dry slag is still rising at this time, indicating that some other valuable elements in the solution are also precipitated with precipitation, which may lead to the increase of zinc loss rate. Therefore, considering the above situation, the reaction time of 120min is the most appropriate.

3.2 Generation of precipitation

According to the experimental principle, when the low valence arsenic in the solution is oxidized by iron ions, the next step is to gradually form precipitation by adjusting the pH of the solution, which is also the most critical step in the chemical precipitation method. The conditions for the formation of precipitation can be met by adding an appropriate amount of neutralizer. The neutralizer baking dust used in this test is the product of roasting the dry residue obtained from the leaching of zinc gold ore, which is different from the traditional acid-base neutralizer. Therefore, it is necessary to explore its relationship with the final arsenic removal effect in the process of adjusting the pH.

3.2.1 Critical value of solution pH

Since the main valuable element involved in this test is zinc, the premise of removing arsenic impurities is to minimize the loss of zinc in the solution. In this experiment, the main direction of zinc loss was the formation of $Zn(OH)_2$ precipitates. Therefore, its generation in the solution should be avoided as much as possible to further reduce the amount of dry residue. From the references and related ionic thermodynamics, it can be known that when the pH value of the solution reaches 6[30-31], the Zn^{2+} in the solution will start to form a large amount of $Zn(OH)_2$, which greatly increases the loss of zinc and the amount of dry residue. Therefore, the final solution of pH should not exceed 6.

3.2.2 Effects of baking dust dosage on arsenic removal

Take 500ml of leaching solution, set the temperature of constant temperature water bath to 70 ℃, after the water temperature rises to the specified temperature, turn on the ultrasonic generator, set its power to 240W and inject oxygen, the oxygen flow is 0.8L/min, add 5-9g baking dust at the same time, adjust the final pH of the solution, and the total reaction time was 90min. Investigate the influence of the amount of baking dust on the arsenic removal effect. The results are shown in Figure 7.

It can be seen from Figure 7 that with the increasing amount of baking dust, the arsenic removal rate has been increasing. However, the increasing rate continues to decrease. When the amount of baking dust is about 7g, the increasing range is significantly reduced, but the amount of dry slag is still increasing sharply. When the amount of baking dust reaches 7-9g, the pH value of the solution has reached 5.5-6.0, which is very close to the critical value specified in the test. At this time, zinc has begun to form precipitation gradually, resulting in a further increase in the amount of dry slag. Although the removal rate of arsenic is also increasing, the range is significantly lower than the increase in the amount of dry slag, Therefore, under comprehensive consideration, the dosage of baking dust used in this test is set at about 7g.

3.3 Two-stage arsenic removal

In addition to exploring the impact of various experiment conditions on the final arsenic removal effect, this experiment also found that under the same conditions of other tests, the final arsenic removal rate can be further improved by dividing the test into two stages(See Table 3). The actual operation is to divide the test into two stages according to the time, and add an appropriate amount of baking dust to adjust the pH value in the first and second stage tests respectively. The segmented test is carried out according to the above best conditions: the reaction time is 120min, the reaction temperature is 70 °C, the amount of baking dust is 7g, the ultrasonic power is 240W, and the oxygen flow is 0.8L/min. After the test, the content and distribution ratio of main elements in the solution and precipitation slag are listed in Table 4, and the XRD analysis diagram of precipitation slag is shown in Figure 8.

Table 3 Comparison of results of segmented experiments

segment	Precipitated slag /g	Arsenic removal rate/%
one	6.2856	84.62
Two	6.4755	92.77

Table 4 content and distribution rate of main elements in solution and Precipitated slag

		Company	As	Fe	Zn
Content	solution	g/L	0.125	0.741	106.93
Distribution rate	solution	%	7.225	39.21	97.46
	Precipitated slag	%	92.775	60.79	2.54

It can be seen from Table 4 that only 7.225% of arsenic remains in the solution and 39.21% of iron. The solution still contains 97.46% zinc. The final arsenic removal effect of the solution is good, lower than 200g/L, meeting the requirements of the subsequent electrolysis process. The pH value of the solution at this time is about 5.5, which further meets the electrolysis conditions

The XRD results in Figure 8 show that the main components in the precipitated slag are related phases of zinc, arsenic and iron. Through the analysis of the content of zinc in the solution in Table 4 and the phase analysis of the baked dust, it can be concluded that the zinc element contained in the slag is mainly Derived from baking dust, in the formed arsenic-iron complex phase, the molar ratio of iron to arsenic is greater than 1, indicating that iron adsorbs part of arsenic, which is consistent with the relevant thermodynamic analysis described in the above experimental principle, consistent with the results obtained by Yang [30]et al.

4. Conclusion

(1) Aiming at arsenic, the main impurity element in the germanium precipitation solution of the zinc oxide leaching solution used in this paper, this experiment utilizes its own characteristics of both arsenic and iron, and uses iron salt chemical precipitation method combined with oxidation method to remove arsenic. By controlling the effect of reasonable process conditions, a good arsenic removal effect can be achieved, and part of the iron in the solution can be removed at the same time.

(2) In this experiment, oxygen is used to oxidize arsenic and iron in the solution under the synergistic action of ultrasonic waves to make them reach a high valence state. The experimental results show that this method is feasible, and the final arsenic removal effect reflects the oxidation effect of this method. Compared with traditional oxidants such as H_2O_2, this method has a good cost-saving effect, the use of baking dust as a neutralizer can well avoid the problem of introducing new elements caused by other neutralizers. At the same time, the loss of zinc in the solution can be avoided to a certain extent. Finally, through the phase analysis of the precipitated slag and related thermodynamic knowledge, it can be concluded that after the pH value reaches 5, part of the arsenic is adsorbed by iron and precipitated, which is in line with the experimental principle described in this paper.

(3) When the experimental conditions are: reaction time 120min, reaction temperature 70℃, baking dust volume 7g, ultrasonic power 240W, oxygen flow rate 0.8L/min, the arsenic removal rate can reach 92.77%, the dry slag produced in the experiment is 6.8732g, and the content was reduced to 0.125g/L. According to industrial requirements, the arsenic removal method described in this paper can reduce the arsenic content in the zinc oxide leaching solution after germanium precipitation to 0.1-0.2g/L to meet the requirements of subsequent zinc sulfate electrolysis, and the amount of slag obtained in the final experiment is small, which meets the requirements of The requirement in industry to minimize the amount of slag.

Declarations

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

National Key R&D Program of China 2021YFC2902801 ,Supported by Yunnan Fundamental Research Projects grant NO.202001AS070009 ,Special fund projects of central government guiding local science and technology development 202107AA110002 ,Yunnan Xingdian Talent Support Project- Industrial innovation talents 2019-1096 ,Yunnan Xingdian Talent Support Project- Young talents 2018-73

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Figures

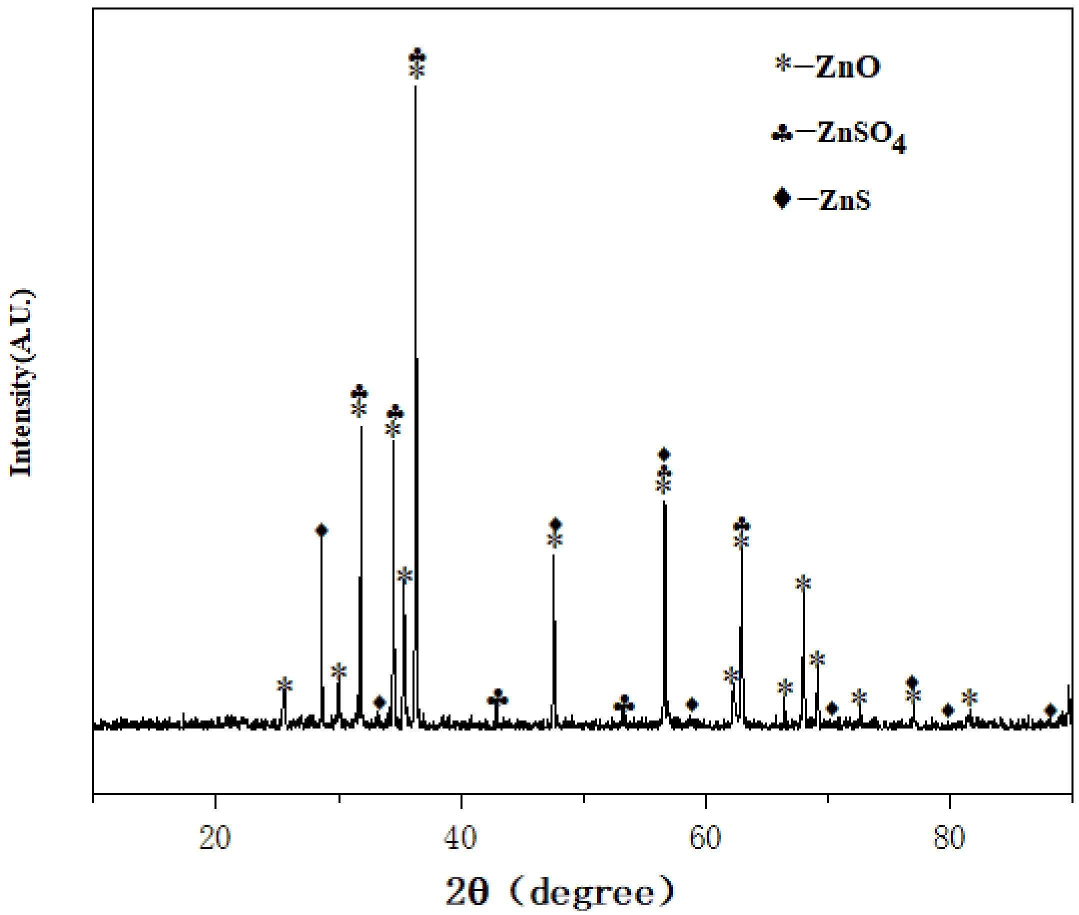

Figure 1

XRD pattern of baking dust

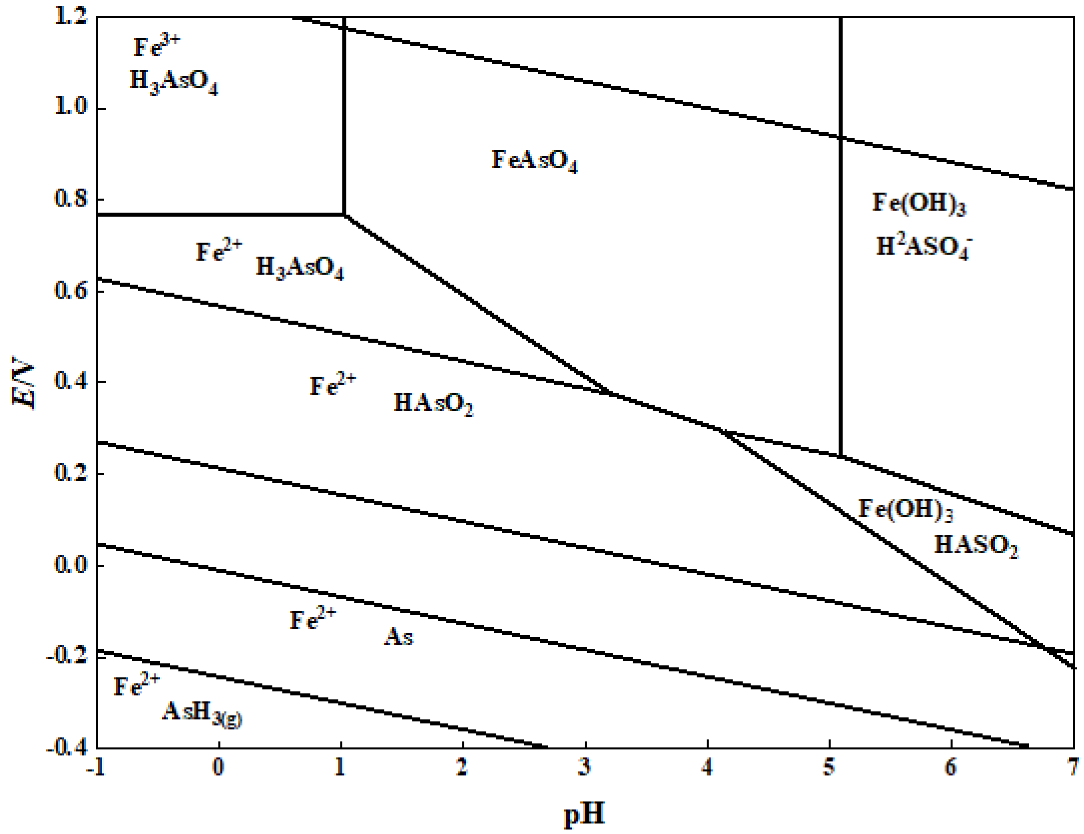

Figure 2

Eh-pH diagram of As-Fe-H2O System at 25 °C

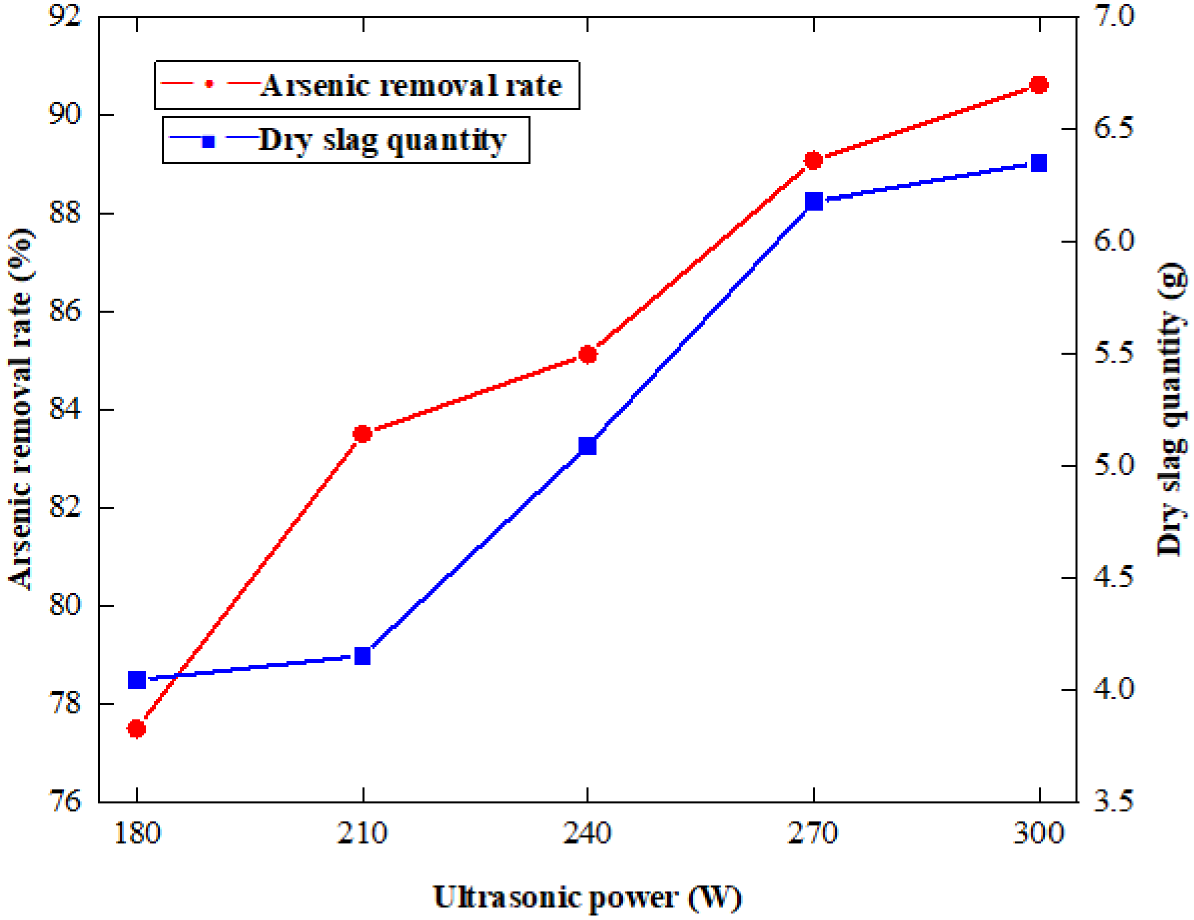

Figure 3

Effect of ultrasonic power on arsenic in solution

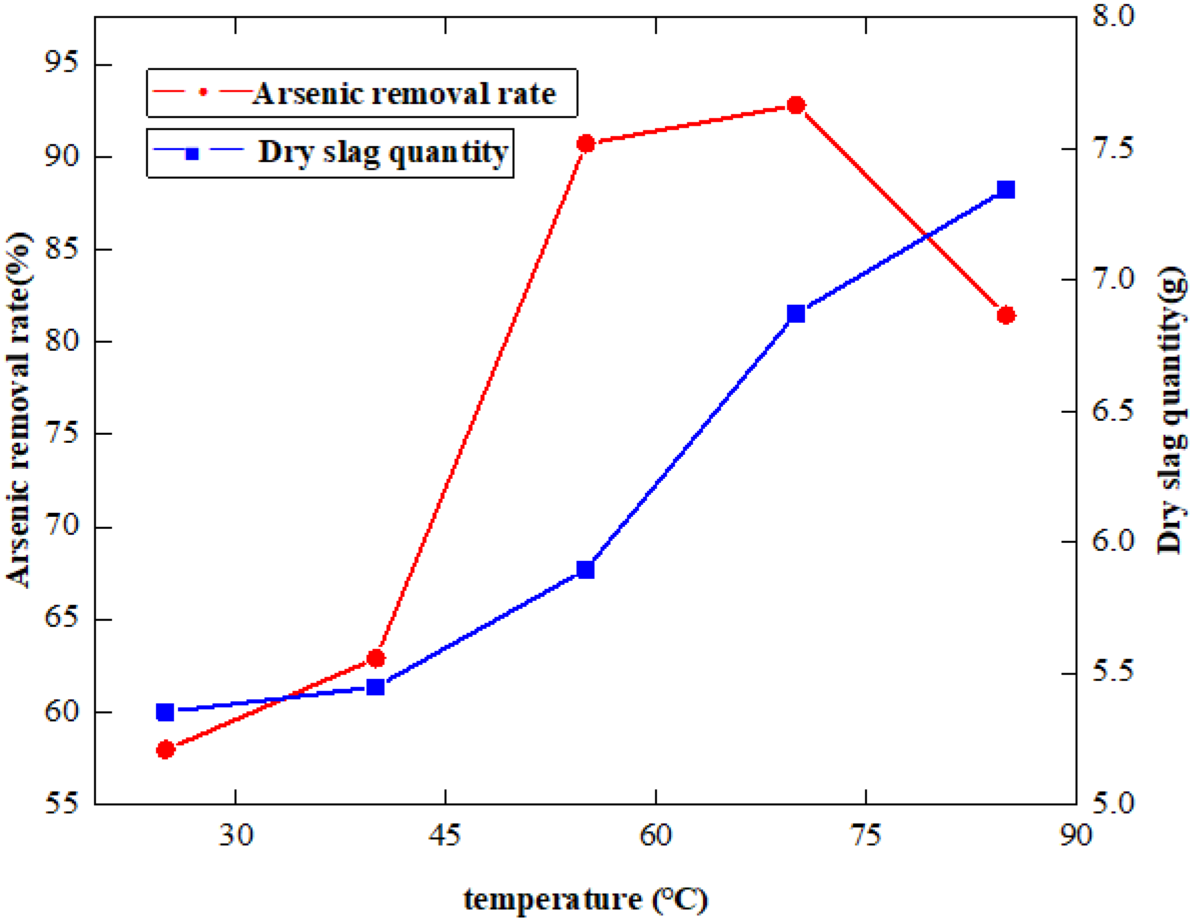

Figure 4

Effect of test temperature on arsenic in solution

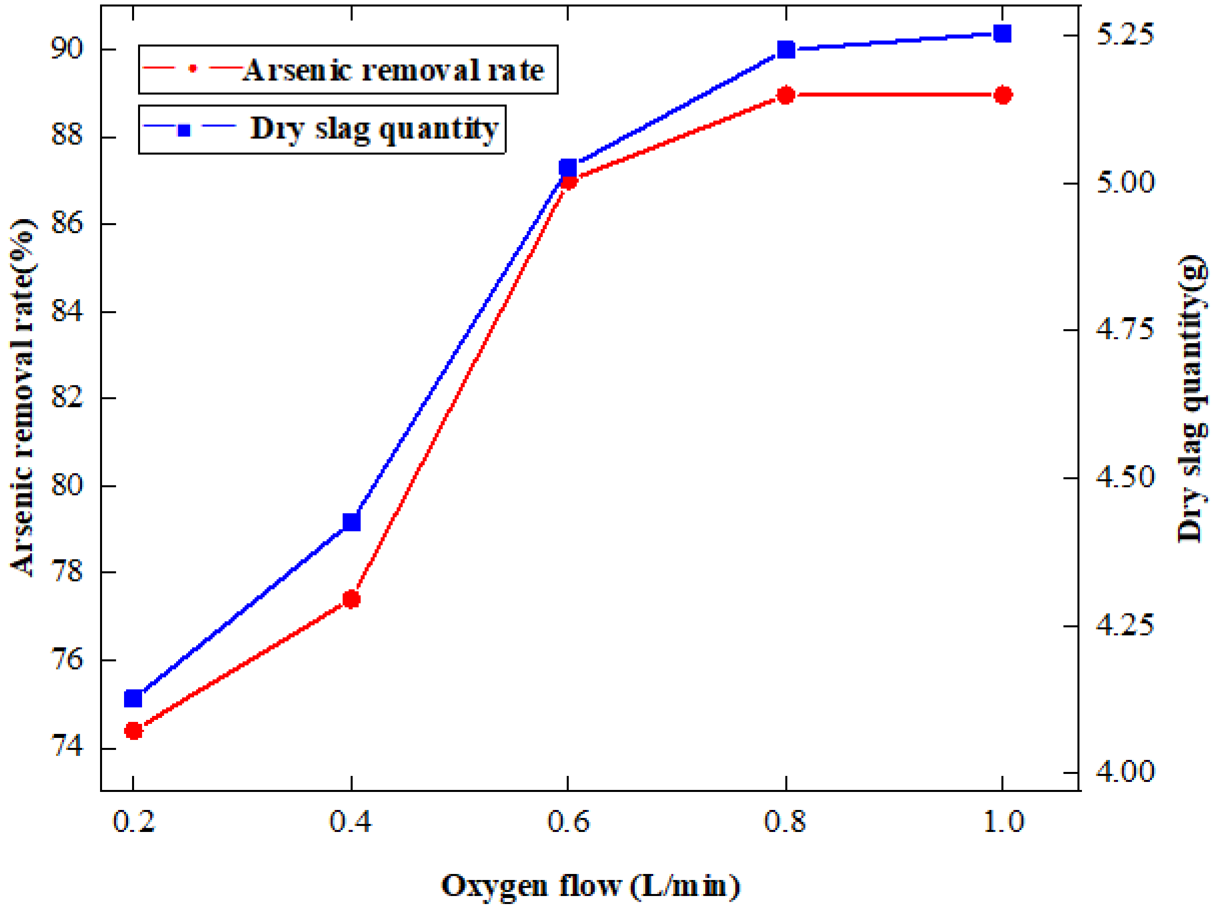

Figure 5

Effect of oxygen flow on arsenic in solution

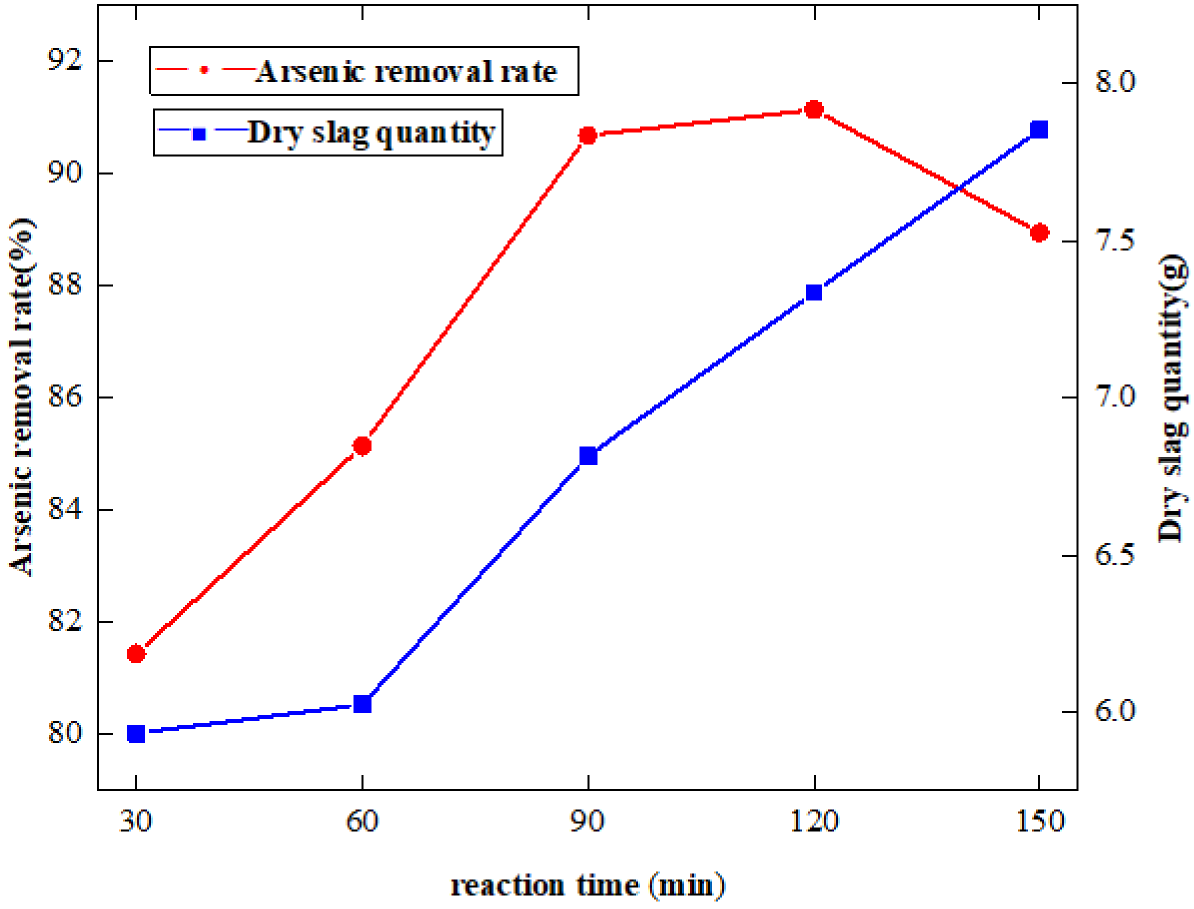

Figure 6

Effect of reaction time on arsenic in solution

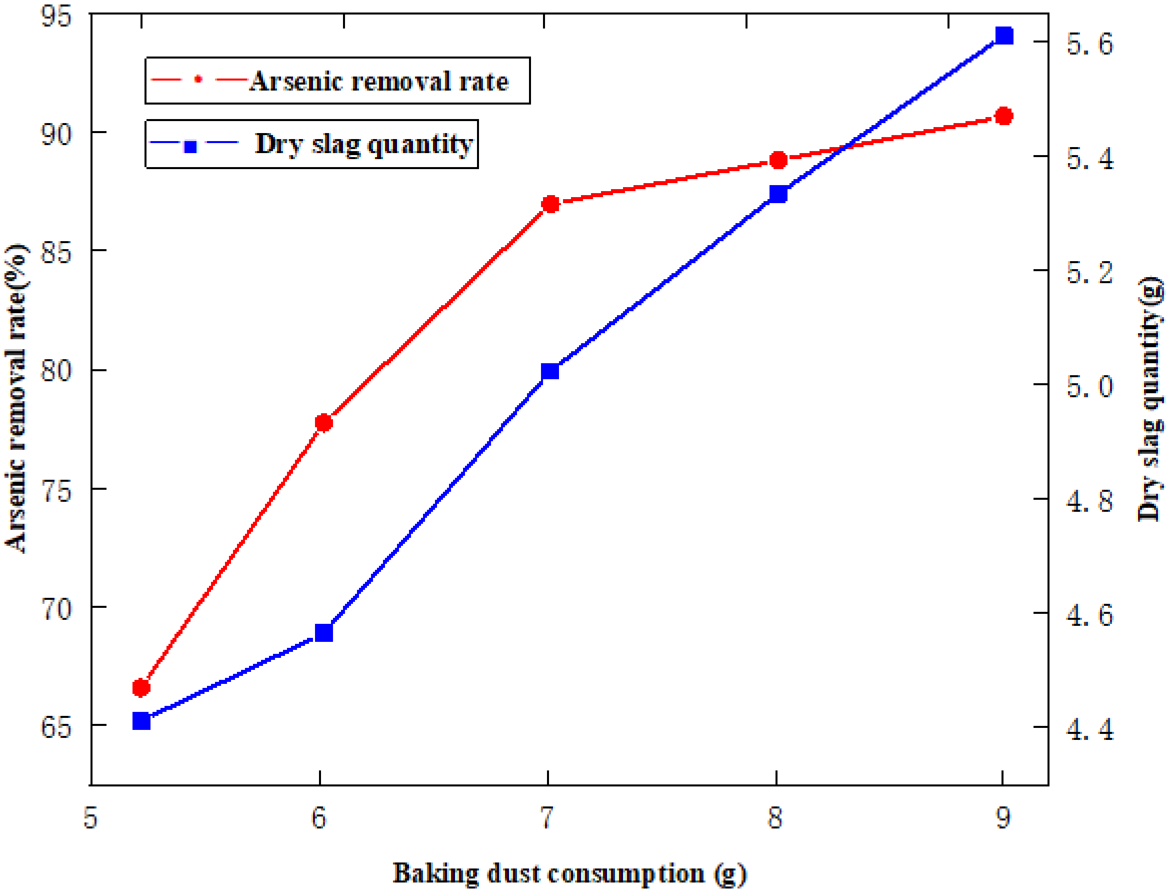

Figure 7

Effect of baking dust dosage on arsenic in solution

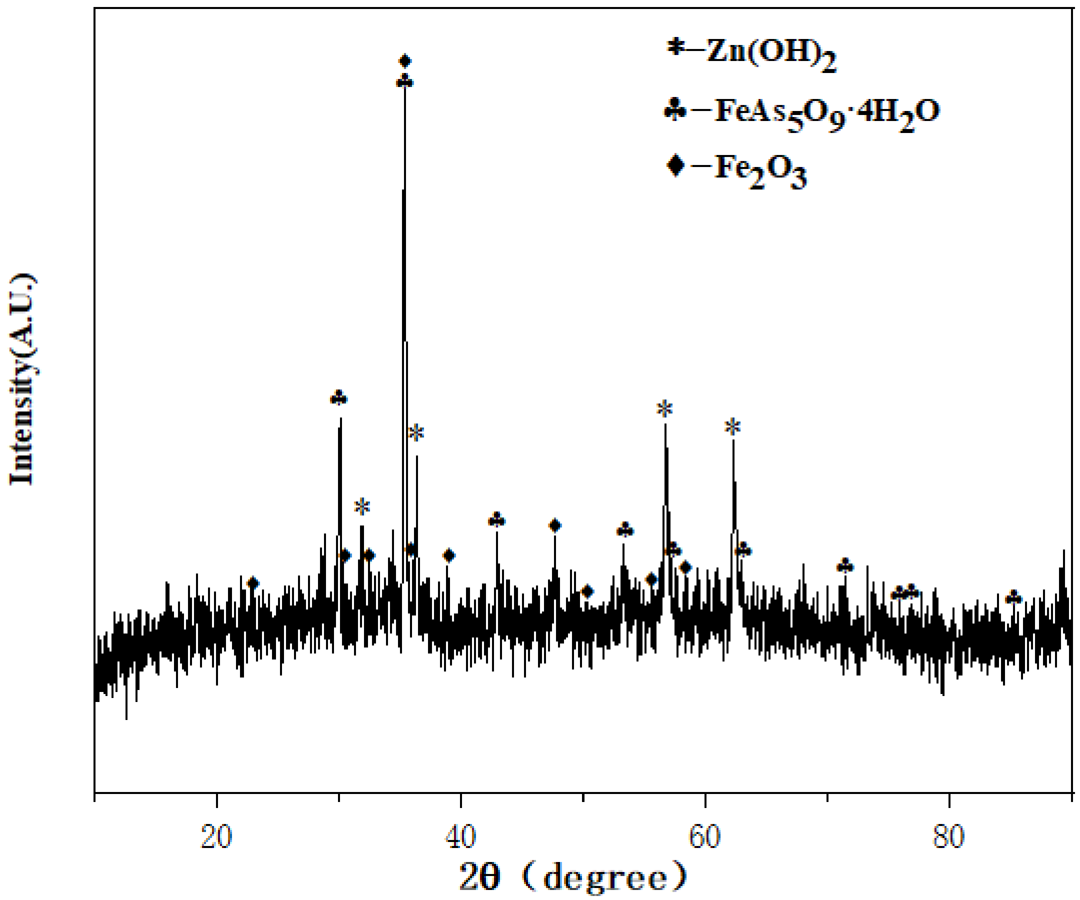

Figure 8

XRD analysis of precipitated slag