



Effects of electrolyte recycling on desulfurization from bauxite water slurry electrolysis

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Abstract: To lower the cost of bauxite electrolysis desulfurization using NaOH solution as the supporting electrolyte, effects of electrolyte recycling on bauxite electrolysis desulfurization were investigated. The results indicate that electrode corrosion, cell voltage, the desulfurization rate and the pH value of the electrolyte have no obvious changes with the increase of cycle times. Additionally, there were some transitive valence S-containing ions in electrolyte after the electrolysis, such as SO_3^{2-} , $\text{S}_2\text{O}_3^{2-}$. However, most of the sulfur in bauxite was eventually oxidized into SO_4^{2-} into the electrolyte, and these S-containing ions did not affect the recycling utilization for electrolyte.

Key words: high sulfur bauxite; electrolysis; desulfurization; electrolyte; recycling

1 Introduction

During alumina production process, S-containing phase is transformed into sulfates (Na_2SO_4) [1], resulting in many problems, such as the decline of dissolution rate of Al_2O_3 , the corrosion of equipment, the reduction of alumina production yield and quality [2]. Once the Na_2SO_4 scale is generated, the operation efficiency of the whole production system and heat transfer coefficient of the equipment will be lowered, and thus increasing the energy consumption [2]. At present, there are many methods for bauxite desulfurization, including calcination [3], flotation [3,4], wet desulfurization [5] and sintering desulfurization with adding reducing agent and so on [6]. But these methods have many limitations, such as low desulfurization rate, harsh reaction condition and complex process, especially for SO_2 emission due to the calcination.

Pyrite, the main S-containing phase in high sulfur bauxite [3], is the same as the S-containing phase in coal. The process of electrolysis desulfurization relies mainly

on the electrochemical oxidation. Pyrite could be oxidized into sulfate by oxidant produced from water electrolysis on anode, achieving the desulfurization through solid–liquid separation. Electrolysis desulfurization has many advantages, including low temperature, normal pressure and high desulfurization ratio [7]. It is noted that electrolysis could also remove simultaneously both organic and inorganic sulfur in minerals, such as coal desulfurization [8]. Additionally, based on the previous results [9–12], the electrolysis desulfurization is more suitable to inorganic ore compared with coal water slurry (CWS) electrolysis [13–16], since anode is covered by organic products as organic ore is electrolyzed.

In our previous work, the feasibility of bauxite electrolysis desulfurization had been verified [17]. To lower the cost of bauxite electrolysis, electrolyte recycling was proposed in this work. However, the process of electrolysis desulfurization might be affected by electrolyte recycling. With the increase of the electrolyte cycle times, the S-containing anions in electrolyte are accumulated gradually. It might bring

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about bad effects to the desulfurization reaction, and finally affecting the desulfurization efficiency. However, if there are no effects of electrolyte recycling on electrolysis desulfurization, electrolyte recycling could be applied to the desulfurization of bauxite electrolysis, which not only could lower the cost of production, but also could lower the environmental pollutant risk. Thus, it is very essential to study the effects of electrolyte recycling on bauxite electrolysis desulfurization.

In this work, the optimal experiment was not done for the ore sample, and thus the best experimental conditions were not obtained. However, this research was focused on the feasibility of electrolyte recycling. It did not affect the experimental results. Grinded bauxite was prepared into slurry with NaOH solution, and the effects of electrolyte recycling on desulfurization from bauxite electrolysis were studied through a variety of characterization methods. Meanwhile, the desulfurization mechanism of bauxite electrolysis in NaOH solution and the conversion of sulfur state in the filtrate were examined by the analysis of the bauxite and the filtrate before and after electrolysis.

2 Experimental

2.1 Samples

A type of high sulfur bauxite from Guizhou province of China was selected as sample. Bauxite composition was analyzed by X-ray fluorescence (XRF, AXIOS, Holland), as listed in Table 1, and ore phase was characterized by X-ray diffraction (XRD, X' Pert PRO MPD, Holland), as shown in Fig. 1. Figure 2 shows the particle distribution of bauxite. As can be seen from

Table 1 Composition of bauxite sample (mass fraction, %)

Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	TiO ₂	Others	Total sulfur
39.93	29.11	10.7	9.48	3.68	7.1	3.95

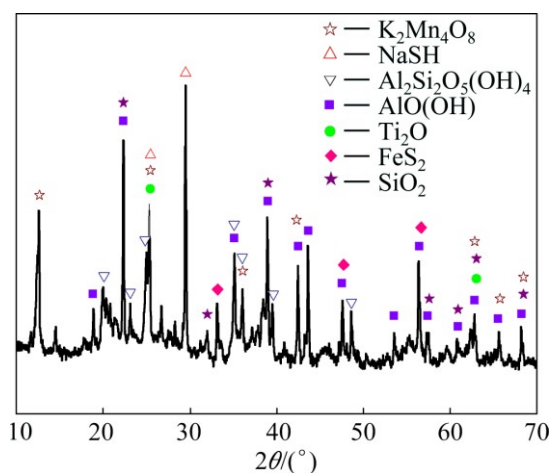


Fig. 1 XRD pattern of bauxite

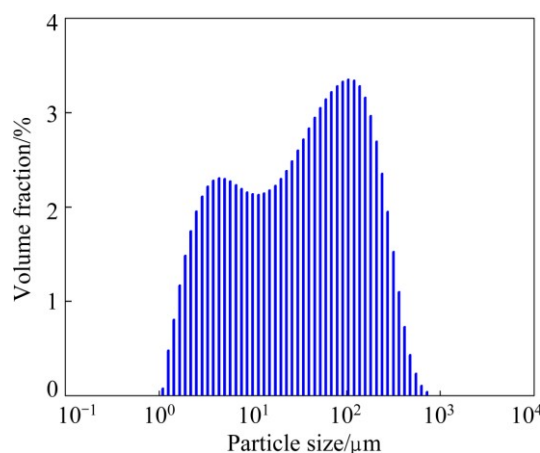


Fig. 2 Particle size distribution of bauxite

the data, the aluminum in bauxite mainly exists as the form of diaspor, and the sulfur mainly exists as the form of pyrite. Additionally, there are also potassium manganate and other impurities. The total sulfur content in bauxite is 3.95%.

All chemical compounds used in this work were listed as follows: NaOH solution (1.0 mol/L), dilute hydrochloric acid solution (1.0 mol/L, 3 mol/L), BaCl₂ solution (10%), Pb(Ac)₂ solution (1 mol/L), HNO₃ solution (2 mol/L), AgNO₃ solution (0.5 mol/L), ethanol (95%), Na₂CO₃ solution (100 g/L), ZnSO₄ solution ((ZnSO₄·7H₂O) 100 g/L), glacial acetic acid (1 + 10), iodine standard solution (0.1 mol/L), starch indicator (5 g/L) and deionized water.

2.2 Experiment

The experimental apparatus is shown in Fig. 3, including DC-3006 circulating water bath and WYK-3030S DC power supply. The active area of the nickel electrode was 4 cm × 4 cm. Electrolysis conditions were as follows: the current of 2.4 A, the electrolysis temperature of 90 °C, the electrolyte volume of 400 mL, the slurry concentration of 25 g/L, the stirring speed of 500 r/min, the concentration of NaOH of 1.0 mol/L and the electrolysis time of 4 h. After electrolysis, the filtrate was retained by suction filtration. 1.0 mol/L NaOH was added to the filtrate in each experiment until 400 mL, and then it was reused as the electrolyte. The filter residue was washed repeatedly until there is no white precipitate generated by adding the BaCl₂ solution acidified with hydrochloric acid, and then it was dried. Changes of the electrolysis voltage under the cycle constant current and the pH of the electrolyte before and after the reaction were recorded. Bauxite phase before and after electrolysis was analyzed by XRD, and the sulfur contents in bauxite before and after electrolysis were analyzed by C-S analyzer (LECO CS-344, USA). The sulfur states in the filtrate were

tested by titration, and the crystal of the last filtrate was analyzed by XRD.

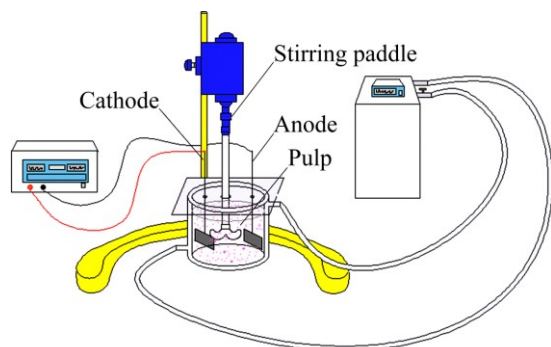


Fig. 3 Schematic diagram of electrolytic device

2.3 Desulfurization rate calculation

The method was focused on the sulfur content in the bauxite after electrolysis [17]. If considering the mass variation of bauxite, the desulfurization rate was high. Whereas the sulfur content in the bauxite after electrolysis was still high, thus the computing method was as follows:

$$D = \frac{w_1 - w_2}{w_1} \times 100\% \quad (1)$$

where D is the desulfurization rate; w_1 and w_2 are the mass fractions of sulfur in bauxite before and after electrolysis, respectively.

3 Results and discussion

3.1 Effects of electrolyte recycling on corrosion and cell voltage

Figure 4 shows the effects of electrolyte recycling on electrode. From Fig. 4, certain amount of the dark matter is deposited on cathode, and there is a little change of electrode with the increase of cycle times. There is red matter on anode, but there is no obvious corrosion on anode. As far as the corrosion of the electrode is concerned, the electrolyte can be recycled. During the bauxite electrolysis process, the anode lost electrons, and the metal ions entered into electrolyte. Additionally, FeS_2 was oxidized into Fe^{3+} and SO_4^{2-} by oxidants on the surface of anode. Most of the Fe^{3+} was generated $\text{Fe}(\text{OH})_3$, which would become Fe_2O_3 further, because of the instability of $\text{Fe}(\text{OH})_3$ [18–20]. Then, the red oxidation film, Fe_2O_3 , was generated covering on the surface of the anode. A small quantity of Fe^{3+} or Ni^{2+} was reduced on cathode, and forming metallic iron or nickel (alloy).

Figure 5 shows the changes in cell voltage with cycle times. The cell voltage has a fluctuation after the rapid decline, subsequently, being steady with time.

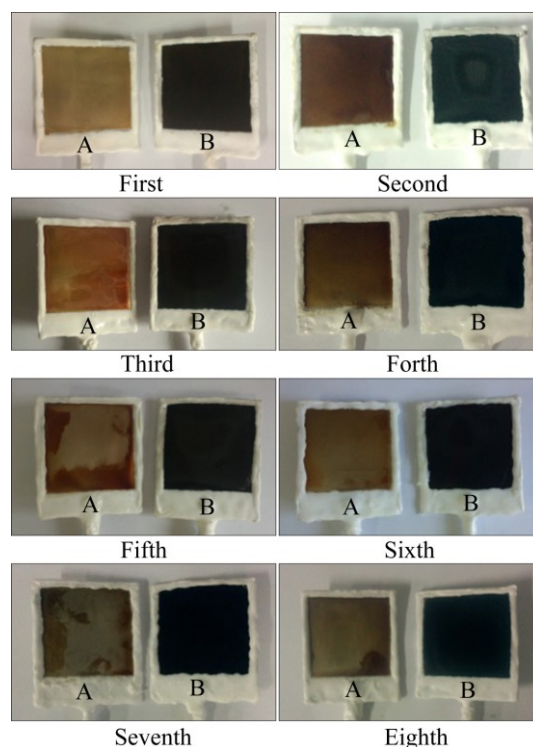


Fig. 4 Effects of electrolyte used times on electrode (1 mol/L of NaOH; 25 g/L of BWS concentration; 0.15 A/cm² of current density; A—Anode; B—Cathode)

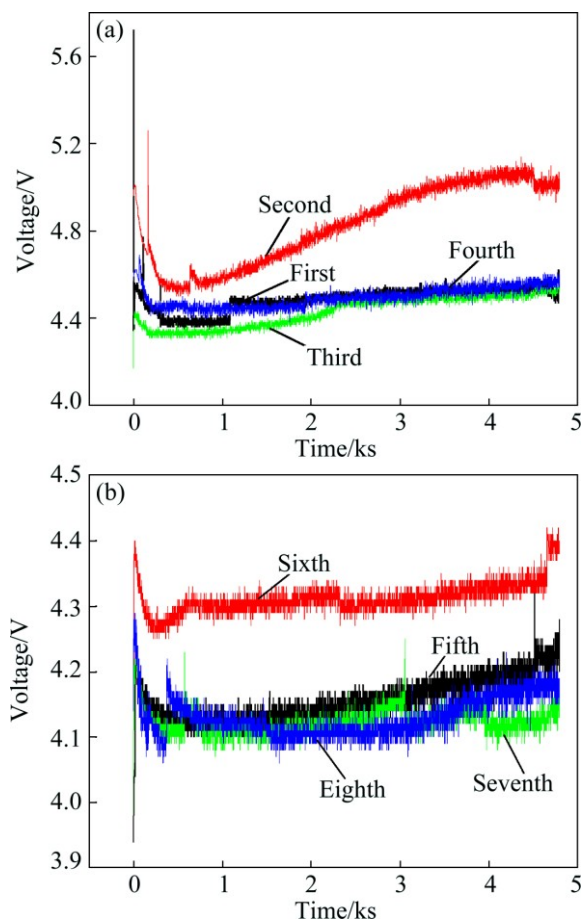


Fig. 5 Effects of electrolyte used times on cell voltage

In NaOH solution, the polarization process of nickel electrode resulted in rapid decrease in cell voltage [21]. From Fig. 5, with the increase of electrolyte cycle times, the cell voltage varies between 4 and 5 V, and there is no obvious increase or decrease. The change in the average voltage with electrolyte recycling is shown in Fig. 6. As can be seen from Fig. 6, the average voltage shows a slight downward trend with the increase of the electrolyte cycle times, resulting from the increase in ion concentration with electrolyte recycling, such as SO_4^{2-} . The lower the voltage is, the smaller the power energy consumption is. Therefore, the electrolyte recycling is beneficial to saving power energy for bauxite electrolysis.

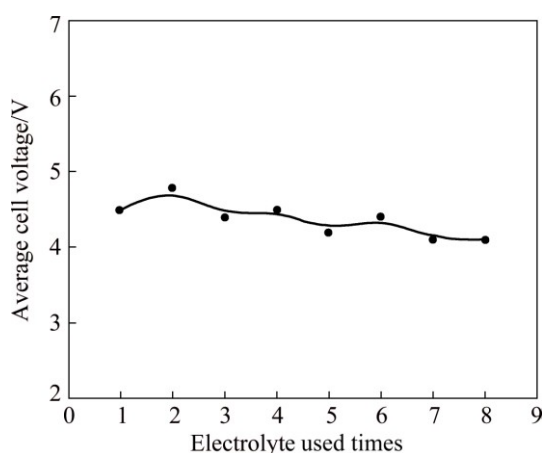


Fig. 6 Effects of electrolyte used times on average cell voltage

3.2 Changes of desulfurization rate and pH

Desulfurization rate is an important indicator for evaluating the effect of electrolyte recycling. The increase of electrolyte cycle times could effectively save the production cost under the premise of good desulfurization effects. From Fig. 7, the desulfurization rate has some changes with the increase of the electrolyte cycle times after it drops to 10% from 20% for the first time. The electrolyte recycling not only saves NaOH

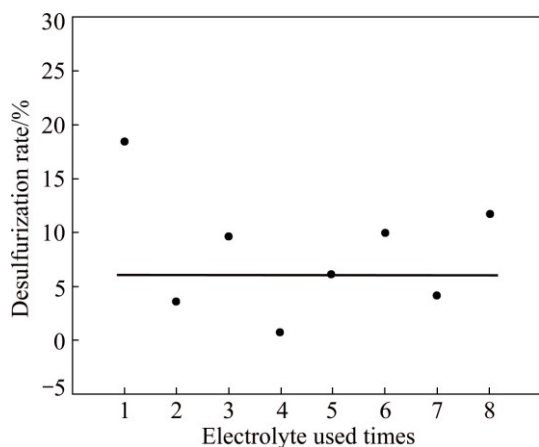


Fig. 7 Effects of electrolyte used times on desulfurization rate

solution, but also lowers environmental pollutant. Because the electrolysis condition is not the best, the desulfurization rate is low. However, the above experimental conditions do not influence the research on electrolyte recycling.

Figure 8 shows the changes in the pH value with electrolyte cycle times. As we know, the pH value of 1.0 mol/L NaOH was 14. The pH value of electrolyte dropped to 13.21 after the first electrolysis, indicating that part of NaOH was consumed. The pH value of the electrolyte should be slightly less than 14, because 1.0 mol/L of NaOH was added into the electrolyte until 400 mL after each reaction. The change of pH value of the electrolyte after reaction is not obvious with the increase of the electrolyte cycle times. This indicates that part of NaOH solution has been consumed during bauxite electrolysis process. For NaOH system, FeS_2 was oxidized into Fe^{3+} and SO_4^{2-} . But, there were not lots of Fe^{3+} in NaOH solution, thus OH^- in solution reacted with Fe^{3+} , generating $\text{Fe}(\text{OH})_3$ sediment, and resulting in the consumption of OH^- .

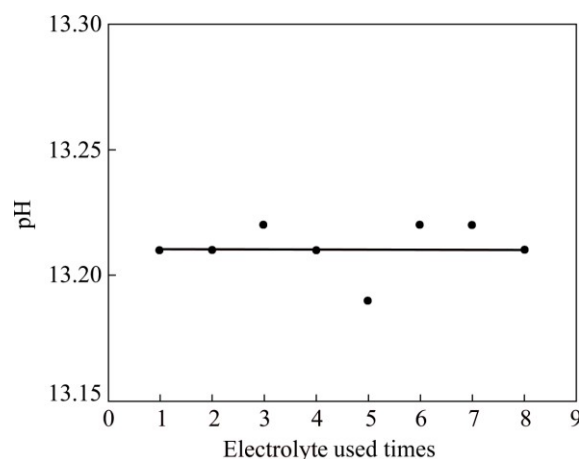


Fig. 8 Effects of electrolyte used times on pH

3.3 Phase change and sulfur form in electrolyte

Figure 9 shows the XRD patterns of bauxite after the first and the eighth electrolysis. From Fig. 9, there are mainly $\text{AlO}(\text{OH})$ peaks and impurities peaks containing SiO_2 and TiO_2 in bauxite after electrolysis. Moreover, there are $\text{Fe}(\text{OH})_3$ peaks which are the product of the electrolysis oxidation process of FeS_2 . The result indicates that the method of electrolysis oxidation could effectively remove S-containing phase in bauxite.

Figure 10 shows the XRD patterns of electrolyte crystal after the first and the eighth electrolysis. There are NaOH peaks in the electrolyte crystal after the first electrolysis. The main component of crystals is NaOH, but there is hydrate of NaOH peaks in the crystals due to the solid of NaOH absorbing water soon and becoming damp in the air. Meanwhile, there are sulfate peaks, such as K_2SO_4 and Na_2SO_4 , also thiosulfate peaks, such as

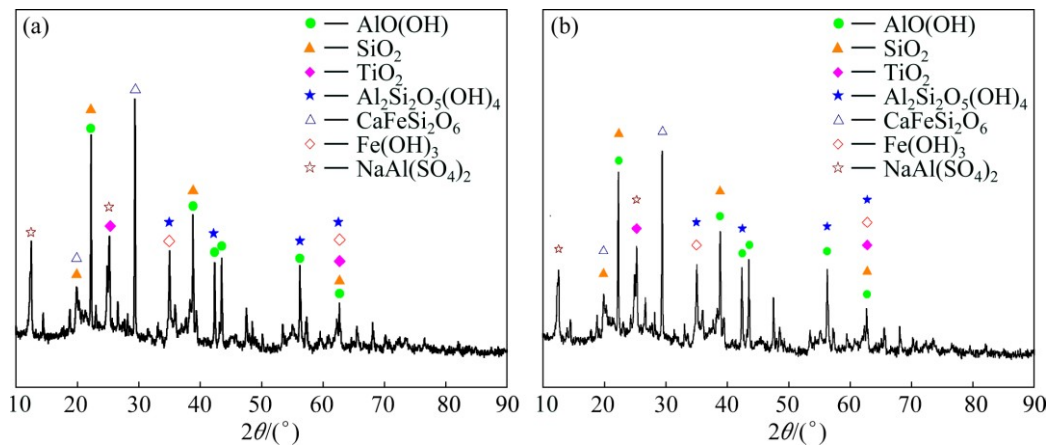


Fig. 9 XRD patterns of bauxite after the first (a) and the eighth (b) electrolysis

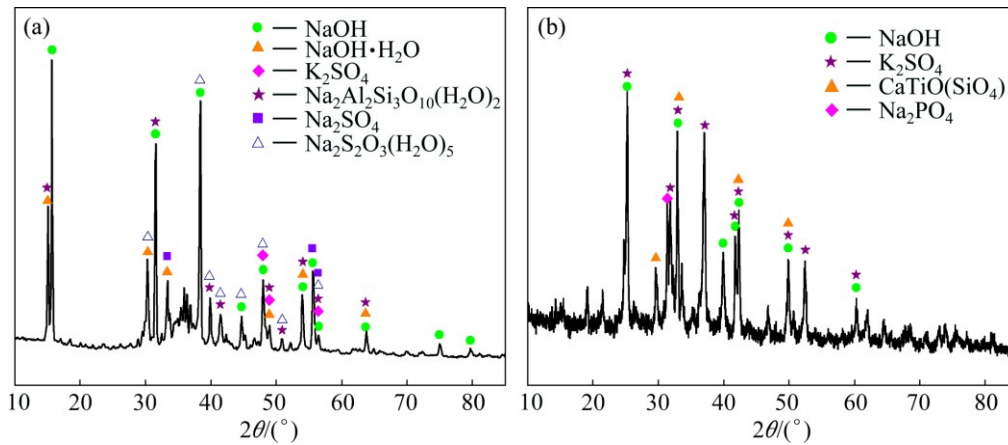


Fig. 10 XRD patterns of electrolyte crystal after the first (a) and eighth (b) electrolysis

$\text{Na}_2\text{S}_2\text{O}_3(\text{H}_2\text{O})_5$. This indicates that the oxidation process of FeS_2 underwent complex changes during bauxite electrolysis process. The conversion of FeS_2 was not directly converted from solid phase sulfur into liquid phase sulfur (SO_4^{2-}). There are also NaOH peaks in electrolyte crystal after the eighth electrolysis, and the main component of crystals is still NaOH . The sulfate was found, such as K_2SO_4 , in the electrolyte crystals, indicating that FeS_2 eventually could be oxidized into SO_4^{2-} for bauxite electrolysis.

The last (eighth) filtrate was tested by titration, and the results are shown in Table 2. The white precipitate was generated by adding the BaCl_2 solution acidified with hydrochloric acid. This proves that there was SO_4^{2-} in the electrolyte solution and FeS_2 eventually could be oxidized into SO_4^{2-} by oxidants generated from water electrolysis. There was little change after adding the $\text{Pb}(\text{Ac})_2$ solution dropwise into the filtrate. There was no black precipitate generated, so there was no S^{2-} in the filtrate. There were no significant changes in the filtrate after adding the AgNO_3 solution nitrated dropwise, so there was no $\text{S}_2\text{O}_3^{2-}$ in the filtrate. The SO_3^{2-} in the filtrate was titrated by GB 10500–2009 [22]. A

Table 2 Sulfur states in last electrolyte

Object	Method	Phenomenon	Result
SO_4^{2-}	Adding the BaCl_2 solution acidified with hydrochloric acid dropwise	Generate white precipitate insoluble in acid	Existence
S^{2-}	Adding the $\text{Pb}(\text{Ac})_2$ solution dropwise	Nothing	Inexistence
$\text{S}_2\text{O}_3^{2-}$	Adding AgNO_3 solution nitrated dropwise	Nothing	Inexistence
SO_3^{2-}	GB 10500–2009	Consume a drop of iodine standard solution until test solution turned blue	Inexistence

drop of iodine standard solution was consumed until the test solution turned blue, indicating that there was almost no SO_3^{2-} in the filtrate. SO_3^{2-} would easily be oxidized to SO_4^{2-} by air, so it could not be detected from the filtrate.

GU et al [23] found that the oxidation reaction of

pyrite was divided into two steps, including the oxidation of pyrite to S^0 and the oxidation of S^0 to SO_4^{2-} . According to the results above, it can be inferred that the oxidation process from S^- of solid phase in bauxite to SO_4^{2-} of liquid phase is a chain reaction process [24,25]. There were some S-containing ions from S^- oxidation, such as S^0 , SO_3^{2-} and $S_2O_3^{2-}$ [26]. Finally, they were oxidized into SO_4^{2-} further. SO_3^{2-} was easy to be oxidized into SO_4^{2-} or $S_2O_3^{2-}$ by reacting with the elemental sulfur (S^0) in the solution. In the initial stage, due to the high concentration of S^- in the solution, the speeds of SO_3^{2-} generated and consumed were both faster in the solution. The reactions could keep dynamic balance, and the concentration of SO_3^{2-} remained relatively unchanged. However, the concentration of S^- was continuously reduced in the later stage. The speed of SO_3^{2-} generated was reduced, so its concentration decreased with time. The concentration of $S_2O_3^{2-}$ rapidly increased with time until reaching the maximum concentration, and then gradually decreased. But the production process of $S_2O_3^{2-}$ was a fast reaction process, the $S_2O_3^{2-}$ concentration remained at a high level. While the reaction process which was further oxidized into SO_4^{2-} was slow. Thus, the main product was SO_4^{2-} in the first filtrate after a long time reaction, followed $S_2O_3^{2-}$, almost no $S_2O_3^{2-}$. When the cycle times increased, the $S_2O_3^{2-}$ was eventually oxidized into SO_4^{2-} through the complex changes after the eighth time.

AHLBERG et al [9] suggested that the pyrite oxidation mainly generated sulfate sulfur in alkaline solution. According to the study on the mineral phase evolution of BWS electrolysis [27] and the results above, the electrode reaction for BWS electrolysis in alkaline solution is shown in Fig. 11.

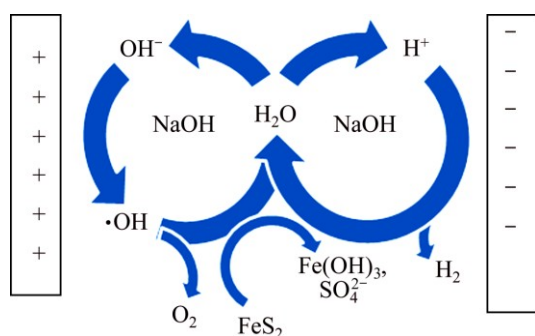
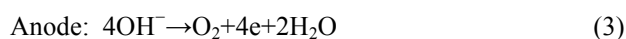
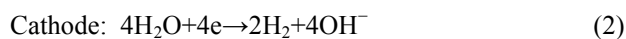
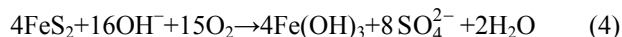


Fig. 11 Desulfurization mechanism from BWS electrolysis in alkaline solution

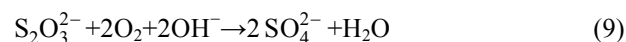
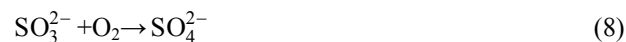
The main reactions of bauxite electrolysis desulfurization in alkaline solution are as follows:



Liquid:



The conversion of sulfur in the liquid phase is as follows:



Some researches suggested that electrochemical oxidation could produce hydroxyl radical ($HO\cdot$) through Fenton reaction [28–30]. In the electrochemical reaction, some active intermediates could be generated on the surface of electrode, such as $HO\cdot$ and $HO_2\cdot$ [31]. These intermediates involved in oxidation reaction [32], especially for hydroxyl radical ($HO\cdot$) [33]. Thus, the reaction mechanism of desulfurization from bauxite electrolysis in alkaline solution could be speculated. In alkaline solution, a number of strong oxidants were generated on anode from water electrolysis [31], such as $HO\cdot$ and $HO_2\cdot$. Some oxidants formed oxygen gas due to their reaction, and others reacted with pyrite producing $Fe(OH)_3$. Finally, most of the sulfur in bauxite was oxidized into SO_4^{2-} through the complicated change in valence.

4 Conclusions

1) There is no obvious corrosion on the surface of electrode with electrolyte recycling. The change in pH of the electrolyte is not obvious with the increase of the cycle times of the electrolyte.

2) The recycling of electrolyte hardly influences desulfurization from BWS electrolysis. However, the electrolyte should be supplemented after each cycle due to NaOH consumption.

3) There are transitive valence S-containing ions in the electrolyte after recycling. Finally, they are oxidized into SO_4^{2-} .

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电解液循环对铝土矿电解脱硫的影响

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摘 要: 为了降低电解脱硫成本, 采用 NaOH 溶液作为电解液, 研究电解液循环对铝土矿电解过程的影响。研究发现随着电解液循环次数增多, 电极腐蚀程度、电解电压、脱硫率及母液 pH 值均无明显变化。另外, 电解后电解液中含有一些过渡态的含硫离子, 如 S₂O₃²⁻ 和 SO₃²⁻, 最终被氧化为 SO₄²⁻ 溶于水中, 这些离子的生成不影响电解液的回收利用。

关键词: 高硫铝土矿; 电解; 脱硫; 电解液; 循环

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