RESEARCH USING ELECTROLYSIS METHOD TO SEPARATE IRON FROM QUY XA ORE IN LAO CAI PROVINCE

NGHIÊN CỨU SỬ DỤNG PHƯƠNG PHÁP ĐIỆN PHÂN ĐỂ TÁCH KIM LOẠI SẮT KHỎI QUẶNG QUÝ XA LÀO CAI

Ha Manh Chien*, Luong Viet Cuong

Viet Tri University of Industry, Vietnam

*Corresponding author: hamanhchienbachkhoa@gmail.com

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Abstract - In this paper, the research process of separating iron from Quy Xa ore in Lao Cai province has been investigated by electrolysis method. The ore is soaked in H_2SO_4 acid solution to separate iron ions, this solution is then filtered to remove insoluble substances and electrolyzed to precipitate iron. Component analysis by Energy-dispersive X-ray spectroscopy (EDX), weight analysis was used electronic scales, check the surface by scanning electron microscope (SEM). The results showed that high purity iron has precipitated. The maximum amount of iron precipitated when the current density was 4.2 A/dm^2. The amount of iron precipitate increases with increasing electrolysis time. The surface structure was small fine particles tightly packed when using surfactants concentration of Sodium Dodecyl Sulphate (SDS) 0.02 g/l.

Key words - Điện phân; quặng Quý Xa; mật độ dòng; cấu trúc bề mặt.

Tóm tắt - Trong bài báo này, nghiên cứu tách sắt từ quặng Quý Xa tỉnh Lào Cai bằng phương pháp điện phân đã được thực hiện. Quặng được ngâm trong dung dịch axit H_2SO_4 để tách ra các ion sắt, dung dịch này sau đó được lọc bỏ chất không tan rồi tiến hành điện phân để kết tủa sắt. Phân tích thành phần bằng phương pháp phổ tán xạ tia X (EDX), khối lượng được xác định bằng cân điện tử, kiểm tra bề mặt bằng phương pháp hiển vi điện tử quét (SEM). Kết quả chỉ ra là đã kết tủa được sắt với độ tinh khiết cao. Lượng sắt kết tủa lớn khi thực hiện ở mật độ dòng 4,2 A/dm^2. Lượng sắt kết tủa tăng nếu tăng thời gian điện phân. Cấu trúc bề mặt là những hạt nhỏ khi sử dụng chất hoạt động bề mặt Sodium Dodecyl Sulphate (SDS) 0,02 g/l.

Từ khóa - Electrolysis; Quy Xa ore; current density; surface structure.

1. Introduction

Iron is one of the most common metals and plays a crucial role in various industries such as construction, mechanical engineering, and transportation. Due to its high demand, research into efficient methods for recovering and producing iron from ore sources is essential. The traditional method for iron recovery is pyrometallurgy, primarily through the reduction of iron ore using coke in blast furnaces. However, there are significant limitations, including the consumption of large amounts of fossil fuels, substantial CO_2 emissions causing the greenhouse effect, and the risk of environmental pollution, which severely affects human health and ecosystems [1–3].

Given these drawbacks, the trend of researching environmentally friendly alternative methods has attracted increasing attention. Among these, electrolysis has emerged as a promising approach. With the development of renewable energy sources such as solar and wind power, the energy cost for electrolysis has decreased significantly, opening up economic possibilities for metal recovery from ores. Electrolysis has been successfully applied to recover metals such as zinc, copper, and tin, owing to advantages such as closed processes, recyclable electrolyte solutions, and minimal generation of hazardous waste.

Several studies have employed electrolysis to precipitate iron, including the separation of iron from clay and kaolin [1], precipitation of iron in glycol solvents [4, 5], and precipitation of iron and iron alloys in environments containing $CaCl_2$ [6]. However, there are few published works on the use of electrolysis to separate iron from Quy Xa ore in Lao Cai. In this paper, we present the application of the electrolysis method to separate iron metal from Quy Xa ore in Lao Cai. The ore is soaked in H_2SO_4 solution to extract iron, followed by electrolysis of the solution to precipitate iron ions as metallic iron. This study aims to evaluate the practical applicability of the electrolysis method in processing domestic iron ores, contributing to minimizing environmental impacts compared to traditional metallurgical methods.

2. Research methods

2.1. Materials and chemicals

Finely ground Quy Xa iron ore from Lao Cai. Hydrochloric acid (HCl), sulfuric acid (H_2SO_4), and Sodium Dodecyl Sulphate with purity greater than 99%, produced in China.

2.2. Equipment

The research utilized an RH basic KT/C magnetic stirrer manufactured by IKA, Germany. The stirring bar is 3 cm long and 0.5 cm in diameter. Chinese filter paper, Der ee DE-960 TR multimeter, Digital Multimeter DT9205A. Glass beakers of 100 ml, 200 ml, and 1000 ml capacity. Titanium electrodes measuring 18 cm in length and 5 cm in width, vacuum pump, vacuum funnel, vacuum flask. The DC power supply used was model BC-1815 by LiOA, Vietnam, with adjustable DC voltage from 2–18 V and a maximum current of 15 A. A pH meter by Mettler Toledo, Switzerland, was used.

The analytical balance used was manufactured by Denver, Germany, with measurement capability from 0.0001 g to 220 g and a resolution of d = 0.0001 g.

2.3. Experimental procedure

Weigh 18 g of Quy Xa iron ore and add to 200 ml of H_2SO_4 solution at a concentration of 180 g/l, stir thoroughly using a magnetic stirrer for 2 hours, then continue soaking the ore for an additional 22 hours. The solution is then filtered to remove insoluble components. Analysis determined the concentration of iron ions in the solution to be 48.8 g/l (iron was analyzed by titration; iron ions in the solution were fully oxidized to Fe^{3+} using HNO_3 combined with heating, then determined by titration with H_2Ssal indicator). Next, dilute the filtered solution with distilled water at a 1:1 volume ratio, adjust the pH of the solution to 1–2, and proceed with electrolysis. The cathode used for iron precipitation is made of copper-zinc alloy, with a precipitation area of 3.8 × 3.3 cm. The anode is made of titanium. Electrolysis is conducted at ambient temperature (25–28°C).

The effect of current density was investigated under conditions of 0.5, 1.5, 3.2, 4.2, 5.5, and 12.2 A/dm², with an electrolysis time of 3 hours.

The effect of electrolysis time on the amount of iron precipitated was studied at a current density of 1.5 A/dm², with electrolysis times of 3, 6, and 9 hours.

The effect of the surfactant SDS was investigated under electrolysis conditions of 1.5 A/dm² current density, 3 hours electrolysis time, and other conditions as previously described. In addition, the electrolyte solution was supplemented with Sodium Dodecyl Sulphate at concentrations of 0.0, 0.02, 0.04, and 0.06 g/l. Electrolysis time was set to 1 hour [7].

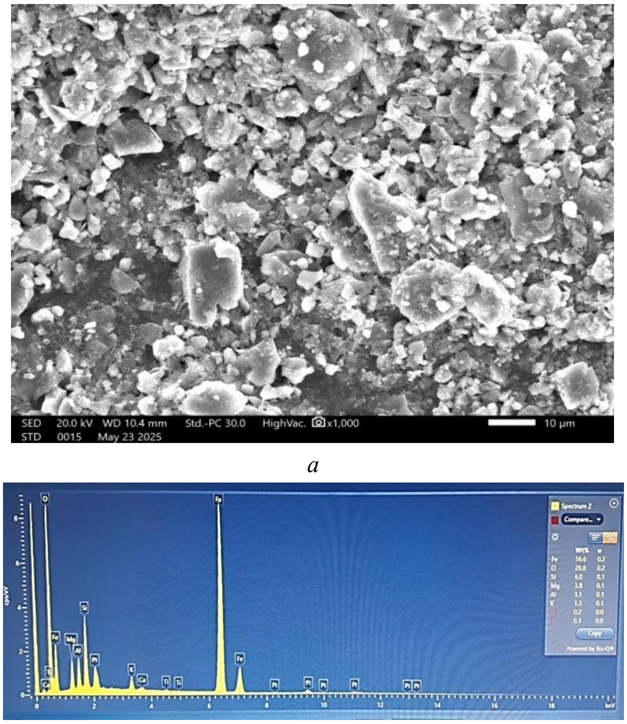
3. Research results and discussion

3.1. Determination of size and composition of raw Quy Xa Iron ore in Lao Cai

Figure 1 shows the images of the ore particles and the EDX analysis spectrum of the ore. The results indicate that the ore particles have a size of approximately 0.1–10 µm. The mass composition of the iron ore includes 56.6% Fe, 28.8% O, 6% Si, 3.8% Mg, 3.1% Al, and 1.3% K. The imaging results show that the iron ore particles are finely ground, with no strong interparticle bonding observed.

3.2. Images and composition of the precipitated layer

To evaluate the formation and characteristics of the iron precipitate layer after electrolysis, the surface of the electrode was observed using an optical camera and the chemical composition was analyzed by Energy-dispersive X-ray spectroscopy (EDX). The images of the copper alloy substrate and the iron precipitate layer are shown in Figure 2. The optical images clearly show the formation of an iron precipitate layer on the electrode surface after 3 hours of electrolysis at a current density of 1.5 A/dm². Compared to the initial copper-zinc alloy substrate (before electrolysis), the post-electrolysis surface exhibits a new material layer with distinctly different structure, indicating that precipitation has occurred.

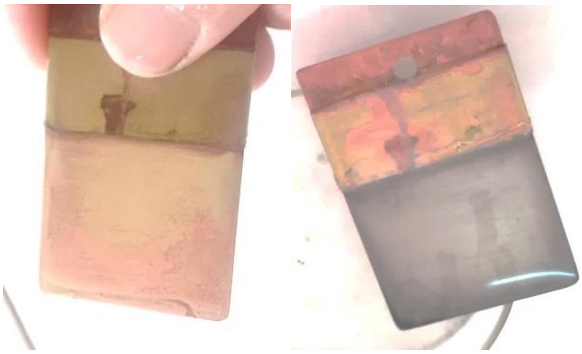


	Wt%	σ
Fe	56.6	0.2
O	28.8	0.2
Si	6.0	0.1
Mg	3.8	0.1
Al	3.1	0.1
K	1.3	0.1
[illegible]	0.2	0.0
[illegible]	0.1	0.0

a

b

Figure 1. *SEM images and EDS spectrum analysis of iron ore particles. a, SEM image of ore particles; b, EDX spectrum analysis of ore particles*



a *b*

Figure 2. *Images of copper alloy substrate and iron precipitate layer. a, Image of copper alloy substrate; b, Image of iron precipitate layer after 3 hours of electrolysis at 1.5 A/dm²*

EDX analysis of the iron precipitate layer is presented in Figure 3. The EDX analysis of the precipitate layer clearly shows the presence of Fe as the main component. The analysis revealed Fe, Cu, and Zn elements in the sample. The appearance of these elements is explained by the formation of iron through electrochemical reactions during electrolysis. The Cu and Zn metals are present because the substrate is made of copper-zinc alloy; during EDX imaging, electrons penetrated through the iron precipitate layer and interacted with the substrate, resulting in the detection of these metals in the spectrum. However, the signal peaks for Cu and Zn are quite low, indicating that the iron precipitate layer is relatively thick. Based on the ore composition and EDX results, it can be concluded that the precipitate layer consists mainly of iron. This is reasonable since, according to the composition of the starting material, aside from iron ions, only metal ions such as Al^{3+}, Mg^{2+}, and K^+ could be present in the leaching solution. These ions have highly negative redox potentials ($E^o_{Al3+/Al}$ = - 1.66; $E^o_{Mg2+/Mg}$ = - 2.37 V; $E^o_{K+/K}$ = - 2.93 V) and thus are not precipitated at the cathode, whereas iron has a higher redox potential and readily precipitates on the electrode ($E^o_{Fe2+/Fe}$ = - 0.44 V). This explains why the precipitate layer obtained contains almost exclusively iron, ensuring high purity.

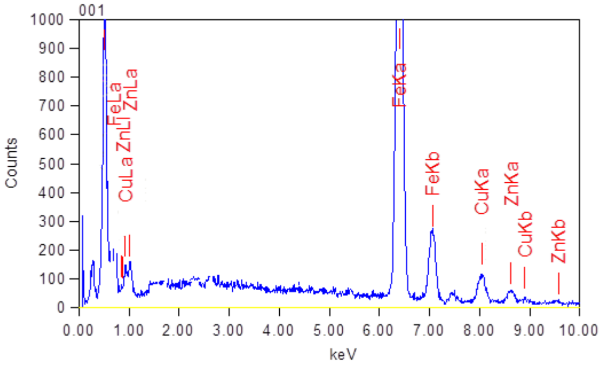


Figure 3. *EDX spectrum analysis of iron precipitate layer*

3.3. Effect of current density on the amount of precipitated iron

The mass of the precipitate layer on samples is shown in Figure 4. The results indicate that at low current density (0.5 A/dm²), the amount of precipitated iron obtained is very small. The reason is that at low polarization potential, the preferential reduction reaction is the reduction of hydrogen ions (H^+ to H_2) rather than Fe^{2+} ions [8], resulting in reduced iron precipitation efficiency.

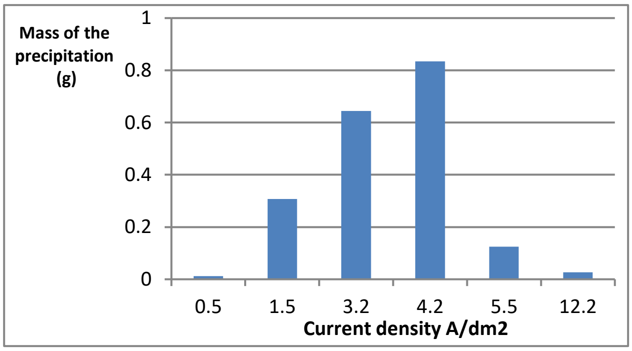


Figure 4. *Effect of current density on the mass of the precipitate layer*

As the current density increases from 1.5 to 4.2 A/dm², the amount of precipitated iron increases significantly. In this range, Fe^{2+} reduction becomes dominant, and the iron layer adheres firmly to the electrode surface, forming a precipitate layer with good adhesion. Notably, at 4.2 A/dm², the precipitate mass reaches its maximum value. However, observations show that the precipitate layer under these conditions is powdery and has poor adhesion. This phenomenon is thought to be due to rapid discharge rates causing iron atoms to crystallize in a disordered manner, leading to a loose structure.

When the current density is further increased to 5.5 and 12.2 A/dm², the amount of precipitated iron decreases, and precipitation occurs in the solution, causing turbidity.

The reason is that the rate of H^+ reduction to H_2 increases sharply, rapidly decreasing the H^+ concentration near the cathode surface. Consequently, local pH increases, favoring the formation of iron hydroxide precipitates ($Fe(OH)_x$), which hinder the direct reduction of Fe^{2+} to metallic iron [9].

Thus, current density has a pronounced effect on both the mass and morphology of the iron precipitate layer, with 4.2 A/dm² identified as the optimal value for achieving maximum precipitation efficiency, although attention should be paid to the structural characteristics of the precipitate layer under these conditions.

3.4. Effect of electrolysis time on the amount of precipitated iron

The measured mass of iron precipitate as a function of electrolysis time is shown in Figure 5. The results indicate that the mass of iron precipitate increases with electrolysis time. Specifically, the precipitate mass after 6 and 9 hours is greater than after 3 hours, reflecting the natural law that prolonged electrolysis leads to more Fe^{2+} ions in solution being reduced to metallic iron and deposited on the electrode. However, when analyzing the growth rate of the precipitate mass over time, it is evident that this rate does not increase sharply but tends to slow down. This suggests that under the experimental condition of low current density (1.5 A/dm²), precipitation is not limited by Fe^{2+} ion concentration (which is relatively high), but is mainly governed by electrode surface kinetics, i.e., the reaction rate at the electrode surface plays a decisive role [10].

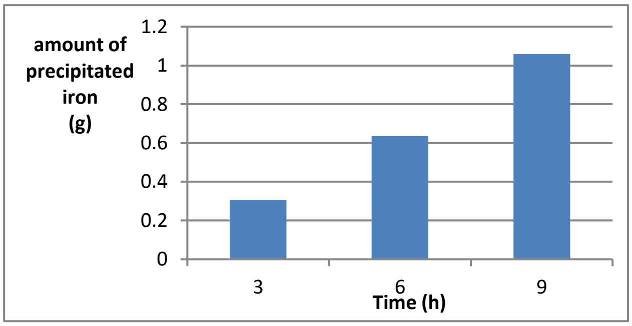


Figure 5. *Effect of electrolysis time on the amount of precipitated iron*

3.5. Effect of surfactant on the surface of the precipitate layer

SEM images of the surface of iron precipitate layers with varying SDS concentrations are shown in Figure 6. The results indicate that the surfactant significantly affects the formation of the precipitate layer. At a surfactant concentration of 0.02 g/l, the surface structure consists of small, tightly packed fine particles. At concentrations of 0.04 and 0.06 g/l, the surface structure comprises larger, more porous particles. This can be explained by the fact that at 0.02 g/l, the surfactant acts to wet the substrate, preventing H_2 gas accumulation and facilitating metal precipitation. At this concentration, the sites of iron precipitation and surfactant adsorption on the substrate alternate, resulting in a fine and compact precipitate layer. At higher concentrations (0.04 and 0.06 g/l), the surfactant occupies more surface sites and is less displaced during iron crystallization [7]. This causes iron to crystallize only at certain positions, leading to a surface structure of larger, more porous particles. Thus, it is evident that SDS concentration directly affects the surface structure of the iron precipitate layer, with 0.02 g/l identified as the optimal concentration for obtaining a fine, homogeneous precipitate layer.

a)

001 30 µm

b)

001 30 µm

c)

001 30 µm

d)

001 30 µm

Figure 6. *SEM images of the surface of iron precipitate layers with different SDS concentrations*
a, without surfactant; *b, with surfactant at 0.02 g/l*; *c, with surfactant at 0.04 g/l*; *d, with surfactant at 0.06 g/l*

4. Conclusion

Electrolysis was conducted to precipitate iron from the leaching solution of Quy Xa iron ore in Lao Cai, and imaging and compositional analysis results show that the precipitate layer consists mainly of iron.

The investigation of the effect of current density revealed that increasing the current density from 0.5 to 4.2 A/dm^2 significantly increased the mass of precipitated iron, reaching a maximum at 4.2 A/dm^2. However, further increasing the current density to 5.5 and 12.2 A/dm^2 reduced the amount of iron precipitate due to the formation of iron hydroxide precipitates. The effect of electrolysis time was also clearly demonstrated: the mass of iron precipitate increased with electrolysis time (3, 6, 9 hours), indicating that iron recovery depends linearly on time under the experimental conditions. However, the rate of increase in precipitate mass tended to slow down, reflecting the influence of electrode surface kinetics. Additionally, studies showed that the surfactant SDS strongly affects the structure of the precipitate layer. At a concentration of 0.02 g/l, the iron layer obtained had a fine, uniform particle structure. At higher concentrations (0.04 and 0.06 g/l), the structure became more porous and less compact due to the effect of SDS on the crystallization process. Overall, the results demonstrate that the electrolysis of the leaching solution from Quy Xa iron ore can produce an iron precipitate layer, providing an important basis for developing technology to recover iron from domestic ore sources.

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