Efficient Recovery of the Combined Copper Resources from Copper Oxide Bearing Limonite Ore by Magnetic Separation and Leaching Technology

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Abstract: The reserve of the copper-oxide-bearing limonite ore (COBL ore) in Yulong Copper Co., Ltd. is up to 20 million tons with 1.79% of copper content. The characters of the copper resources in the COBL ore are high-proportioned oxidation state (99.98%) and combined state (84.83%). The combined copper oxide is mainly copper-oxide-bearing limonite, which has a copper content of more than 78%. Because of the high altitude and average annual temperature of 15 °C in Tibet, fire leaching cannot be adopted. The leaching efficiency of copper from COBL ore using direct leaching of sulfuric acid is only 40%, which is greatly influenced by temperature and time. Based on the characteristics of COBL ore, a novel combined method of magnetic separation and individual leaching has been proposed to efficiently recover copper resources. Experimental results show that the magnetic concentrates and tailings were obtained by magnetic separation of COBL ore at 0.6 T with the yields were 59.65% and 40.35%, respectively. Due to the obvious leaching properties difference of the magnetic concentrates and tailings, individual leaching process routes were used to treat them. The magnetic concentrate was leached with stirring for 3 days at room temperature (20 °C), and the magnetic tailing was easily leached for 4 h at 40 °C. The recovery efficiency of total copper was 72%, which was about 32% higher than that of the single leaching of the COBL ore. The method proposed in this study achieves environmentally friendly, low energy consumption, and efficient extraction of refractory copper oxide ore.

Keywords: copper-oxide-bearing limonite; combined copper-oxide; magnetic separation; countercurrent leaching; complex mineral resources

1. Introduction

Copper is one of the earliest discovered and most widely used metals. It has been used in defense, aerospace, construction, electrical appliances, transportation and other fields. The main recyclable copper resources are copper sulfide and copper oxide, which are important natural metal resources [1,2]. With the rapid development of the world economy, the demand for copper is increasing, and there is a serious shortage of easily recoverable and high-quality copper resources [3,4]. In recent years, copper sulfide ores have become less and less, and the mining and utilization of copper sulfide ore and free oxidized copper ore are difficult to meet the demand. Thereby, the disposal of combined copper oxide ores has become increasingly prominent. The efficient and reasonable utilization of refractory copper-oxide resources can enhance the utilization of resources, and achieve economic benefits [5].

The Yulong Copper Mine is located in Tibet, China, which has the second largest copper metal reserves in China, at an average altitude of 4500 m. It has abundant copper resources and is suitable for large-scale open-pit mining. However, the copper-oxide-bearing limonite ore (COBL ore) accounts for 77% of copper oxide ore. The COBL ore has
complex properties, fine embedded particle size, and high oxidation ratio and combine ratio, which makes copper extraction difficult. The most common method is sulfide flotation for copper-oxide separation in industrial production [6–8]. However, the copper recovery efficiency of the conventional sulfide flotation process is only 13%, and the direct acid leaching process has very high acid consumption. Moreover, the copper leaching efficiency is only about 40%, and it is difficult to efficiently recover copper resources in the COBL ore. Therefore, it is urgent to develop an efficient and feasible new method for comprehensive recovery and utilization of Yulong COBL ore resources.

Magnetic separation is one of the most widely used and mature technologies, such as removing impurities from mineral concentrates, alleviating the toxicity of wastewater, and separating and purifying biomolecules from biological traits [9–13]. In addition, it is also a simple, cost-saving and environmentally friendly method of separating magnetic and non-magnetic materials [14–16]. It plays a role in separating and pre-desliming of COBL ore. Therefore, magnetic separation can be helpful for the subsequent leaching of the COBL ore.

Hydrometallurgy is a technique that uses solution chemistry to extract valuable metals from ores, such as acid leaching, and alkaline leaching. A lot of hydrometallurgical routes have emerged as potential methods for recovering metals such as nickel, copper, and zinc from low-grade oxide ores and wastes [17–20]. Recycling the heavy metals by acid leaching is widely used [21–23]. Sulfuric acid leaching has a broad application prospect due to its advantages of high copper leaching efficiency, less environmental pollution, and low energy consumption [24]. The COBL ore is a typical complex copper oxide from Yulong Copper Mine, and its copper-bearing minerals are also terribly complicated. At present, there are many studies on the leaching process or leaching efficiency of complex copper oxide ores [25,26]. Leaching efficiency indicates the degree of leaching of the metal to be extracted, that is, the percentage of the metal being leached. However, sulfuric acid leaching alone cannot efficiently recover copper resources from COBL ore.

In this work, a novel magnetic separation–leaching method was proposed to recover copper resources from the complex and refractory COBL ore. The magnetic concentrate and tailing were obtained by magnetic separation. There were obvious differences in their leaching performance. Therefore, magnetic concentrate and tailing were leached at room temperature and 40 °C, respectively. This method effectively utilizes copper resources, saves energy, and reduces consumption. Finally, the difficulty of economic and efficient utilization of copper resources in Yulong iron–copper ore was effectively solved.

2. Experiments
2.1. Materials and Reagent

The COBL ore used in this study were obtained from Yulong copper company located in Tibet, China. It originated from the “Three Rivers” Tethys metallogenic belt of copper–iron polymetallic deposits. The chemical composition of COBL ore was tested by X-ray Fluorescence (XRF) and X-Ray Diffractometry (XRD, ARL EQUINOX 3000, Paris, France). The anode material of XRD is copper palladium, and the scan speed and step are 4 degree/min and 0.02 degree, respectively. The specific databases are ICSD minerals, ICSD inorganics and ICSD organics from Jade 6.5 software. The diffraction angle range of XRD is 5~80 degree. The relationship between the spatial orientation of the diffraction line and the crystal structure can be expressed by the Bragg equation:

$$2d \sin \theta = n\lambda$$  \hspace{1cm} (1)

where $d$ is the crystal plane spacing; $n$ is the number of reflection levels; $\theta$ is the grazing angle; $\lambda$ is the wavelength of the X-rays. The X-ray diffraction image of a crystal is essentially a fine and complex transformation of the microstructure of the crystal, and there is a correspondence between the structure of each crystal and its X-ray diffraction pattern. The results are presented in Table 1 and Figure 1. They showed that the COBL ore mainly contained Fe, Si, Al, Cu, and Ca in the form of limonite (Fe$_2$O$_3$·nH$_2$O), kaolinite
(Al₄[Si₄O₁₀](OH)₈), quartz (SiO₂), and calcite (CaCO₃) crystalline phases. The peak height cannot represent the content of the phase, and the crystallization degree of quartz and calcite is high, and the corresponding peaks are also high.

Table 1. Chemical composition analysis of the COBL ore (wt%).

| Elements | O   | Fe  | Si  | Al | Cu | Mn | Ca | S   | Other + H₂O |
|----------|-----|-----|-----|----|----|----|----|-----|-------------|
| Content  | 31.90 | 45.12 | 6.50 | 4.16 | 1.44 | 0.06 | 0.53 | 0.30 | 9.98        |

Figure 1. X-ray diffraction (XRD) pattern of the COBL ore.

Considering the cost and efficiency, the reagent used in the leaching process was 3.06 mol/L sulfuric acid (H₂SO₄). It was purchased from Hunan Huihong Reagent Co., Ltd., Hunan, China. and its purity was 98%. All solutions in whole experiment were prepared with tap water.

2.2. Experimental Design

The COBL ore was first ground to −74 μm. The sample was filtered and dried after grinding. Then the sample was cooled to room temperature and weighed to 100 g. Fourthly, it was sieved with a 200-mesh sieve. Finally, the product under the sieve (−74 μm) was weighed, 73 g, accounting for 73%. Then it was sorted and separated with the help of a high gradient magnetic separator (DLSD, DLS Co., Ltd., Hunan, China) under the magnetic field strength of 0.6 T. Finally, the COBL ore was sorted into a high iron and low copper magnetic concentrate and a low iron and high copper magnetic tailing. The flowsheet is shown in Figure 2. Due to the obvious differences in their wet leaching properties, the separation method of the fractionated leaching process was adopted subsequently. Agitation leaching experiments were carried out in 500 mL glass flasks. Parameters such as temperature, time, acid concentration, and particle size were investigated to determine the optimum leaching conditions. The leaching temperature range is 20 to 80 °C, the sulfuric acid concentration is 150 to 600 kg/t, the leaching time is 4 h to 7 days, and the particle size range is −0.04 mm, accounting for 53% to 87%.
For magnetic concentrates, it was treated by stirring leaching at room temperature. For magnetic tailing, it was leached by sulfuric acid agitation leaching method at 40 °C. During the leaching experiments, the weight of COBL ore was 100 g. The liquid-to-solid ratio (vol/wt) was 3:1. The leaching experiments were carried out in a mechanical stirring water bath (HJ-4S, SB Co., Ltd., Changzhou, China). The effects of temperature, sulfuric acid concentration, particle size, and leaching time on leaching copper from the COBL ore were investigated, respectively. The copper contents of leaching solution were analyzed by inductively coupled plasma—optical emission spectroscopy (ICP-OES). The leaching residue was tested by XRF.

Figure 2. Magnetic separation process flowsheet of COBL ore.

2.3. MLA Measurements

The original purpose of the Mineral Liberation Analyzer (MLA) system concentrated on determining the degree of ore mineral liberation from gangue [27,28]. It is the most advanced automatic quantitative analysis and testing instrument for process mineralogy parameters in the world. The MLA system employs the FEI Quanta multi-purpose scanning electron microscope, and combines the dual-probe high-speed, high-energy X-ray energy spectrometer as the hardware support of the system. The high-resolution BSE imaging system can analyze mineral compositions as small as 0.2 µm. In this work, the MLA (FEI Quanta 600 platform, Hillsboro, OR, USA) was used to obtain the mineral composition of COBL ore, the intercalation relationship, and the occurrence state of copper element, which resulted in quantitative textural data of the natural mineral assemblage in the COBL ore.

3. Results and Discussion

3.1. Process Mineralogy Analysis of COBL Ore

Because the surrounding copper was adsorbed by the limonite and distributed in the form of fine particles on the surface and inside of the limonite, the complex COBL ore was finally formed. The mineral composition of COBL ore is presented in Table 2. It can be seen that the mineral composition of COBL ore is complex, the main mineral is limonite, followed by kaolinite, with a small amount of calcite, plagioclase, chrysocolla, malachite
and quartz. Table 3 indicates the occurrence state of copper element in the COBL ore. As can be seen, the independent copper minerals are mainly malachite (0.61%) and chrysocolla (1.82%). The copper-bearing minerals are mainly limonite, in which copper accounts for more than 78%, and a small amount occurs in kaolinite-clay and pyrolusite. The oxidation ratio of an ore is the percentage of oxidized ore to the total ore. Generally speaking, the oxidation ratio of copper greater than 30% becomes copper oxide ore. The copper in the combined copper minerals is called combined copper, and the percentage of combined copper in the total copper content of copper minerals is called the combination ratio. The calculation formula is as follows:

$$c = \frac{a}{t} \times 100\%$$  \hspace{1cm} (2)

where $c$ is the combination ratio, $a$ is combined copper oxide content, %, $t$ is total copper content of the ore. Since most of the copper elements are present in the limonite in the form of combined state, Tables 2 and 3 indicate that the ore has the characteristics of high oxidation ratio (99.98%) and high combination ratio (84.83%). Figure 3a is an original image, and Figure 3b is a color image. They are both surface scans of the COBL ore from MLA analysis. They indicate that the composition and intercalation relationship of minerals phases. Many limonite exists individually, so that direct leaching consumes a large amount of sulfuric acid. The minerals are embedded in a very fine grain size and have complex mineral components. As a result, it is difficult to recover copper by conventional flotation and leaching.

Table 2. Mineral composition of COBL ore.

| Mineral            | Content (%) |
|--------------------|-------------|
| Limonite           | 82.57       |
| Chrysocolla        | 1.82        |
| Malachite          | 0.61        |
| Kaolinite          | 6.99        |
| Calcite            | 1.83        |
| Quartz             | 1.10        |
| Plagioclase        | 1.44        |
| Orthoclase         | 0.89        |
| Biotite            | 0.46        |
| Jarosite           | 0.79        |
| Pyroxene           | 0.34        |
| Amphibole          | 0.14        |
| Diaspore           | 0.11        |
| Pyrolusite         | 0.10        |
| Biotite-Phlogopite | 0.06        |
| Coronadite         | 0.05        |
| Alunite            | 0.03        |
| Beudantite         | 0.02        |
| Gibbsite           | 0.02        |
| Other              | 0.63        |

The SEM-EDS images in Figure 4 showed the distribution of iron and copper elements in COBL ore. As shown in Figure 4a, it can be seen that the bound copper is mainly copper-bearing limonite. The independent copper-bearing minerals shown in Figure 4b are mainly malachite. Copper is more uniformly dispersed in limonite mainly in the form of ion adsorption and isomorphism, which makes it difficult for copper to be directly leaching. On the other hand, direct leaching of COBL ore consumes a large amount of sulfuric acid, resulting in waste of resources and increased cost. Therefore, new processes and methods are required for efficient leaching of copper from COBL ore.
Table 3. The occurrence state of copper element in the COBL ore.

| Mineral             | Cu Content (%) |
|---------------------|----------------|
| Limonite            | 78.13          |
| Chrysocolla         | 9.52           |
| Malachite           | 5.64           |
| Kaolinite           | 4.42           |
| Pyrolusite          | 1.74           |
| Biotite-Phlogopite  | 0.28           |
| Coronadite          | 0.10           |
| Beudantite          | 0.02           |
| Alunite             | 0.02           |
| Orthoclase          | 0.02           |
| Jarosite            | 0.03           |
| Gibbsite            | 0.03           |
| Tremolite           | 0.02           |
| Delafossite         | 0.01           |
| Chalcopyrite        | 0.01           |
| Other               | 0.01           |

Figure 3. Minerals intercalation relationship images from MLA analysis: (a) original image; (b) color image.

Figure 4. Surface scans of copper and iron elements in COBL ore from SEM-EDS: (a) limonite and kaolinite; (b) limonite and malachite.
3.2. Effect of Leaching Temperature and Time

Higher temperatures enable faster kinetic reactions in chemical systems [29]. In order to study the extraction of copper from the COBL ore at different temperatures, the COBL ore agitation leaching experiments were performed at 20 °C, 40 °C, 60 °C, and 80 °C. The concentration of sulfuric acid was 300 kg/t (3.06 mol/L), the liquid to solid ratio (vol/wt) was 3:1, and the leaching time was 4 h. According to the results in Figure 5, it can be seen that increasing the leaching temperature had a positive effect on both copper and iron leaching. When the temperature was 80 °C, the highest extractions of copper and iron were obtained, 60.11% and 10.85%, respectively. An appropriate increase in temperature can accelerate the COBL ore reactivity with sulfuric acid. For hydrometallurgical processes, the increase in temperature also accelerates the rate of chemical reaction and diffusion, resulting in an increase in the extraction of metals. However, due to the high altitude and average annual temperature of about 15 °C in Tibet, the increase in leaching temperature will also make the cost increase. Moreover, iron is the key element as it directly affects the acid consumption and also it creates major problems in selective recovery of metals from pregnant leach solution [20]. Therefore, the temperature of leaching was controlled within 40 °C.

![Figure 5. Effect of temperature on metal extractions of COBL ore in agitation leaching.](image)

In chemical systems, the increase in reaction time makes the kinetic reaction faster. A series of COBL ore leaching experiments was conducted with different time. During experiments, the sulfuric acid concentration was 300 kg/t, and the leaching temperature were 20 °C and 40 °C, respectively. The results were shown in Figure 6. It can be seen that after a certain leaching time, the leaching values of the COBL ore at 20 °C presented a linear trend with respect to time, rather than a sharp continuous increase. The extraction efficiency of copper was only about 60% when the leaching time was 7 days. On the contrary, the extraction efficiency of copper for leaching one day could reach 60% when the temperature was 40 °C. This indicated that direct leaching of COBL ore did not yield high recovery efficiency of copper, and it would consume a lot of cost and energy.
3.3. Effect of Sulfuric Acid Concentration

In leaching experiments, the initial acid concentration plays an important role in the leaching of valuable metals. A number of experiments were carried out to determine the metal leaching value at different sulfuric acid concentrations. All experiments were performed under the optimal conditions determined in the previous sections.

In Figure 7, it appeared that sulfuric acid concentration did not have a significant effect on extraction values of copper. On the contrary, the leaching efficiency of iron had been increasing with increasing acid concentration. Furthermore, since iron is more reactive than copper, iron consumes acid more easily. In order to reduce the dissolution of iron, the concentration of sulfuric acid should not be too high. As can be seen from Figure 7, the copper leaching efficiency basically reached the maximum at a sulfuric acid concentration of 300 kg/t, which was about 50%. Therefore, the optimum dosage of sulfuric acid in this experiment was 300 kg/t.

**Figure 6.** Effect of time on metal extractions of COBL ore in agitation leaching.

**Figure 7.** Effect of sulfuric acid concentration on metal extractions in agitation leaching.
3.4. Effect of Particle Size

Mineral particle size can be adjusted by grinding fineness. Therefore, the effect of grinding fitness (−0.040 mm) on COBL ore leaching was studied. The experiments were carried out under the optimal conditions determined in the previous sections. The result is shown in Figure 8. It can be seen that as the proportion of −0.040 mm increased from 53% to 87%, the extraction efficiency of copper and iron did not change significantly. Further, it indicated that the grinding fineness at the micron level did not significantly improve the leaching efficiency of bound copper from COBL ore. Considering the leaching index and grinding cost comprehensively, the proportion of particles with a grinding fineness of 0.040 mm was chosen to be 53%.

![Figure 8](image_url)

**Figure 8.** Effect of particle size on metal extractions in agitation leaching.

3.5. Magnetic Separation

In order to enrich copper in COBL ore, it is necessary to pre-magnetic separation. Magnetic field strength is an important influencing factor in magnetic selection. In this section, different magnetic field strengths were investigated. The mineral particle size is −0.074 mm accounting for 75%. The mass proportions of concentrate 1 (K1), concentrate 2 (K2), concentrate 3 (K3) and tailing (X) are 2%, 15%, 27%, and 56%, respectively. The experimental results are shown in Figure 9 under the conditions of 0.08 T, 0.35 T, and 0.6 T. As can be seen, under weak magnetic field strength (magnetic field strength is 0.08 T), magnetic separation can only recover a small part of strong magnetic minerals. As the magnetic field strength increases, the copper grade of the tailings gradually increases and the iron grade gradually decreases. In addition, because copper is present in limonite in a combined state, it can be seen from Figure 10 that the recovery of both iron and copper increases with increasing magnetic field strength for concentrates. For tailing, the recovery of iron decreases while the recovery of copper increases. Finally, high-iron and low-copper concentrates and high-copper and low-iron tailings are formed.
Figure 9. Influence of magnetic field strength on COBL ore during magnetic separation.

Figure 10. The iron and copper recovery from magnetic concentrates and tailing.

The three concentrates were combined to form the final concentrate. Based on the characteristics of magnetic separation concentrate and tailing, it can be seen that there are significant differences in their leaching properties. Therefore, they can be treated separately by different leaching process methods to achieve efficient recovery of copper resources.

3.6. Leaching of Magnetic Concentrate and Tailing

Because of the high altitude, average annual temperature of 15 °C and in order to save costs, the leaching temperature is 20 to 40 °C, the sulfuric acid concentration is 300 kg/t, the leaching time is 4 h to 3 days, and the particle size range is −0.04 mm, accounting for 53%. The leaching process flow is shown in Figure 11. Due to the high iron grade and the low copper grade, the concentrate of magnetic separation was mainly leached for a long time under room temperature conditions. High residual sulfuric acid concentration was obtained at the end of magnetic concentration leaching experiment, so the leaching solution can be used as a leaching mother liquor for the magnetic separation tailing. The
tailing was treated with sulfuric acid agitation leaching method. The liquid to solid ratio (vol/wt) was 3:1, and the concentration of sulfuric acid was 300 kg/t.

For magnetic concentrate, the leaching time was longer because the concentrate was more difficult to leach. As shown in Table 4 and Figure 12a, increasing leaching time had a promoting effect on the extraction of metals. When the leaching time exceeded two days, the extraction efficiency of copper did not change much, and the dissolution of iron would affect the experiment. Therefore, the optimal time for magnetic concentrate in the experiment was 3 days. For magnetic tailing, the leaching temperature was 40 °C. Table 4 showed that the leaching efficiency of copper was about 82% when the leaching time was 4 h. Moreover, it can be seen from Figure 12b that the leaching efficiency of copper increased with the increase in leaching time, but the increasing trend of iron leaching efficiency was more obvious. When the leaching time was 12 h, the leaching efficiency of iron reached 13%. Therefore, the leaching time should not be too long in order to control the extraction of iron. The optimum leaching time for magnetic tailing was selected as 4 h. The total copper recovery efficiency was shown in Table 4. The copper leaching efficiencies of concentrate and tailing cannot be directly added up, and the total copper recovery has to be calculated based on their respective copper recoveries. It indicated that the copper recovery could reach about 72% when the concentrate leaching time was 3 days. Therefore, the copper leaching efficiency can be increased by about 32% using this process.

Table 4. Magnetic concentrate and tailing leaching results.

| Name   | Leaching Time | Leaching Temperature (°C) | Leaching Efficiency (%) Cu | Leaching Efficiency (%) Fe | Total Copper Recovery (%) |
|--------|---------------|----------------------------|-----------------------------|----------------------------|---------------------------|
| Concentrate | 1 d            | 20                        | 53.12                       | 2.05                       | 68.33                     |
|         | 2 d            |                            | 55.39                       | 2.41                       | 69.40                     |
|         | 3 d            |                            | 59.13                       | 3.86                       | 71.57                     |
|         | 4 d            |                            | 61.17                       | 4.98                       | 72.05                     |
| Tailing | 4 h            |                            | 81.65                       | 3.68                       |                           |
Figure 12. Effect of leaching time on metal extractions in agitation leaching: (a) magnetic concentrate; (b) magnetic tailing.

4. Conclusions

The COBL ore has a complex composition and low copper recovery using a single-stage method. The ore has the characteristics of high oxidation ratio (99.98%) and high combination ratio (84.83%), and the combined copper oxide is mainly copper-bearing limonite, and its copper content accounts for more than 78%. A new magnetic-leaching process was proposed. Good results were achieved, and the process has the characteristics of energy saving, environmental protection and flexibility. The advantage of the new process is to introduce magnetic separation to enrich copper and reduce iron. The magnetic concentrate was leached with stirring for 3 days at room temperature, and the copper leaching efficiency could reach about 60%. The magnetic tailing is easily leached for 4 h at 40 °C. The copper leaching efficiency from magnetic tailing was 81.65%, with a total recovery efficiency of 72%. Compared with a single process, the copper recovery efficiency was increased by about 32%. The process can be used as a guideline for the metal extraction of other complex refractory ores.

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