

Article

Separation of Rhenium from Lead-Rich Molybdenite Concentrate via Hydrochloric Acid Leaching Followed by Oxidative Roasting

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Abstract: Lead-rich molybdenite is a typical rhenium-bearing molybdenum resource in China, which has not been efficiently utilized due to its high contents of lead and gangue minerals. In this study, hydrochloric acid was used for preliminarily removing lead and calcite from a lead-rich molybdenite concentrate. Oxidative roasting-ammonia leaching was then carried out for separation of rhenium and extraction of molybdenum. The hydrochloric acid leaching experiments revealed that 93.6% Pb and 97.4% Ca were removed when the leaching was performed at 95 °C for 10 min with HCl concentration of 8 wt. % and liquid-solid ratio of 5 (mL/g). The results of direct oxidative roasting indicated that 89.3% rhenium was volatilized from the raw concentrate after roasting at 600 °C for 120 min in air. In contrast, the rhenium volatilization was enhanced distinctly to 98.0% after the acid-leached concentrate (leaching residue) was roasted at 550 °C for 100 min. By the subsequent ammonia leaching, 91.5% molybdenum was leached out from the calcine produced from oxidative roasting of the acid-leached concentrate, while only 79.3% Mo was leached from the calcine produced by roasting molybdenite concentrate without pretreatment.

Keywords: molybdenite concentrate; rhenium; lead; oxidative roasting; acid leaching

1. Introduction

Rhenium (Re) is an important metal that has been widely used in many fields, such as national defense, petrochemical engineering, aerospace, photovoltaics, solar cells, etc [1]. The global Re resource reached 2.5 million kg with only 48,800 kg produced in 2014. More than half of the production was from Chile, which has more than 1.3 million kg Re in reserves [2]. Rhenium is a rare metal occurring as rheniite (ReS₂) mainly accompanying molybdenite. It was also found in some copper, niobium yttrium iron, platinum and uranium ores. Typically, copper or molybdenum sulfide concentrates via flotation circuits contain 50–100 g/t (ppm) Re [3]. Because of the high value of rhenium, recovering rhenium from molybdenum concentrate has received much attention.

During the roasting process of molybdenite concentrate, rhenium is volatilized and enters the gas phase as Re₂O₇. A small portion may remain with the calcine. There are two types of roasting: oxidative roasting and solidification roasting [4]. The former process recovers molybdenum by leaching the calcine in ammonium hydroxide to produce ammonium molybdate. The contained rhenium is separated and volatilized into dust in the form of oxides (Re₂O₇), which are soluble in water or hydrogen peroxide solutions [5]. In solidification roasting, both rhenium and molybdenum

are retained in calcine in the presence of slaked lime [6] or soda ash [7]. Rhenium is then extracted by leaching the lime-roasted calcine in sulfuric acid or water [6]. A common problem with the solidification roasting method is attributed to the large amount of slaked lime added, which would lower the contents of molybdenum and rhenium and prolong the enrichment period, exerting an unfavorable effect on the recovery of rhenium. Unlike lime-roasting, the molybdenum and rhenium in soda ash-roasted calcine could be leached in water solution [8].

Molybdenum and rhenium can be simultaneously leached by pressure oxidation leaching (POX), which is divided into two types: acid pressure leaching [9–11] and alkaline pressure leaching according to the leaching medium [12]. The use of high pressure and high concentration acid or alkali corroded the equipment severely. These two metals can also be extracted by other approaches, such as electrochemical leaching [13,14], bioleaching [15,16] and microwave-assisted leaching [17], although they have not been utilized commercially. The current industrial practice in the recovery of rhenium from rhenium-bearing solutions was generally conducted through ion exchange or solvent extraction [18–21].

Up to now, oxidative roasting has widely been used around the world by large manufacturers because of its low cost, easy operation and high efficiency [22,23]. However, the formation of molybdate and sulfate due to the existence of sulfides (PbS, FeS, ZnS and Cu₂S) often restrains the recovery of molybdenum [24]. Moreover, the reactions between gangue minerals and molybdenite would result in serious sintering, which restricts the oxidation of molybdenite and volatilization of rhenium [25]. Previous studies mainly focused on the behavior of molybdenum during the roasting [26,27], but few studies explored the behaviors of rhenium and the gangue minerals in favor of separating rhenium from molybdenum. The volatilization behavior of rhenium and roasting characteristics of molybdenum concentrate deserve further attention.

Lead-rich molybdenite is a typical molybdenum resource in Shanxi province of China, which has not been efficiently utilized mainly due to its high lead content [28]. The successful roast-leach process should volatilize rhenium as much as possible but simultaneously reduce the volatilization of molybdenum, leaving a negligible effect on the subsequent ammonium leaching process. In view of the high lead content in the raw material, hydrochloric acid was adopted to initially remove lead and calcite in this study [29]. The effects of leaching parameters on the removal of lead and the separation of rhenium during subsequent oxidative roasting were examined. Eventually, molybdenum was leached from the calcine in the ammonium solution to identify the favorable effect of HCl leaching.

2. Experimental Section

2.1. Materials

The lead-rich molybdenite concentrate sample was obtained from a dressing plant of Shanxi province, China. The size distribution of the sample was 95.2 wt. % of the particles smaller than 0.074 mm. The main chemical composition of the sample is shown in Table 1. The Mo and Re contents were 43.55 wt. % and 321 g/t, respectively, and the lead content was as high as 4.52 wt. %. The XRD pattern shown in Figure 1 indicates that molybdenum mainly existed in the form of molybdenite (MoS₂) and the gangue minerals were galena (PbS), quartz (SiO₂) and a small amount of calcite (CaCO₃).

The gases for oxidative roasting and cooling were mixed O₂/N₂ and N₂ gases, respectively. The purity of these gases was above 99.99%.

Table 1. Main chemical composition of lead-rich molybdenite concentrate/wt. %.

Mo	Re	Fe	SiO ₂	Al ₂ O ₃	CaO	MgO	WO ₃	Cu	Pb	S
43.55	321 g/t	1.39	4.02	0.18	3.27	0.48	1.66	0.03	4.52	32.46

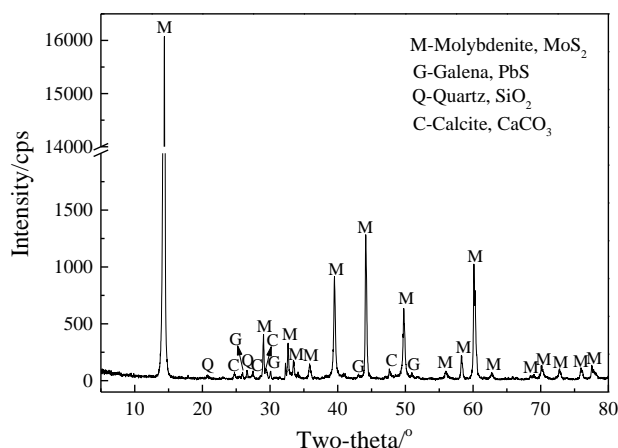


Figure 1. XRD pattern of the lead-rich molybdenum concentrate.

2.2. Experimental Procedure

The experimental procedure mainly included acid leaching, drying, oxidative roasting, cooling, etc. In each leaching test, 5 g of the sample was treated in a beaker containing a predetermined quantity of hydrochloric acid (determined according to the liquid-solid ratio (mL/g)). The beaker was located in a water bath that maintains the leaching temperature with a solution stirred continuously. After leaching for a desired duration, the solution was filtered and the residue obtained was washed with hot distilled water and dried for chemical analysis.

The samples after hydrochloric acid leaching (acid-leached concentrate) were balled into green pellets with 10–14 mm in diameter. The pellets were dried in an oven at 80 °C for 4 h before oxidative roasting. The schematic diagram of the roasting apparatus is shown in Figure 2. The dried pellets were firstly loaded into the reactor (a quartz tube with perforated screen plate at the bottom), into which the nitrogen gas was flushed to expel the air. Then, the quartz tube was transferred into the heating zone of an electric resistance furnace that was preheated at a rate of 10 °C/min to a required temperature. The mixed O₂/N₂ gases were directed into the tube immediately. The composition of oxidative gas was adjusted by the flowmeters of O₂ and N₂, and the total flow rate was fixed at 4.0 L/min to ensure reaction atmosphere. After roasting for a certain time period, the quartz tube was taken out of the furnace and cooled down in N₂. The calcine was then collected for analysis.

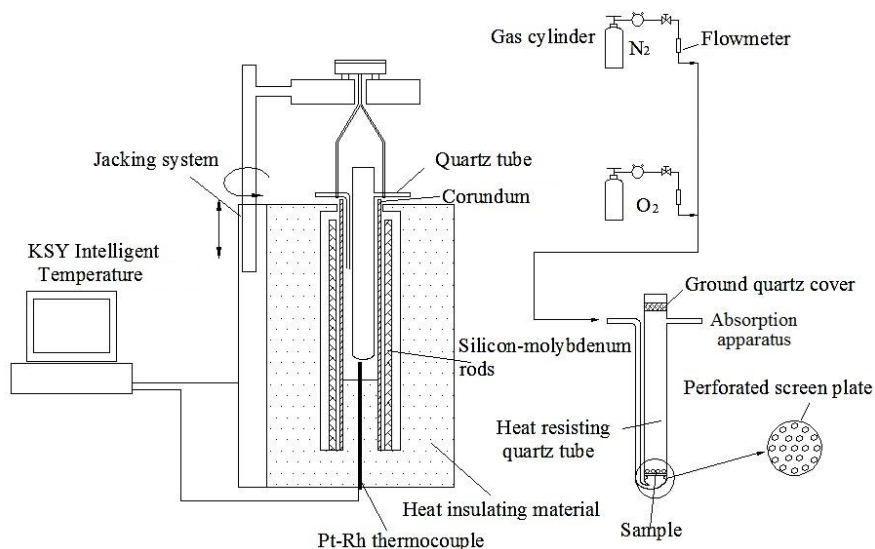


Figure 2. Schematic diagram of the equipment for roasting.

2.3. Analytical Method

The XRD analysis was conducted using a diffractometer (RIGAKU D/Max 2500, Akishima, Japan) under the conditions as follows: Cu K α , tube current and voltage: 250 mA, 40 kV, scanning range: 10°–80° (2 θ), step size: 0.02° (2 θ) and scanning speed: 8°/min.

The contents of Pb and Mo were measured by the chemical titration method, while the Re content was assayed by the instrument of Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES, Thermo Scientific iCAP 7000 series, Waltham, MA, USA).

The acidic leaching ratio of Pb, Mo or Re was calculated by the equation as follows:

$$\beta = \left(1 - \frac{m_1 \times \varepsilon_1}{m_0 \times \varepsilon_0}\right) \times 100\% \quad (1)$$

where β is the leaching ratio, %; m_0 is the weight of molybdenite concentrate, g; ε_0 is the weight fraction of Pb, Mo or Re of molybdenite concentrate, %; m_1 is the weight of leached residue, g; and ε_1 is the Pb, Mo or Re weight fraction of leached residue, %.

The volatilization ratio of Mo or Re was calculated according to the following equation:

$$\alpha = \left(1 - \frac{m_2 \times \varepsilon_2}{m_0 \times \varepsilon_0}\right) \times 100\% \quad (2)$$

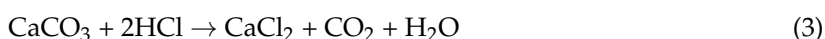
where α is the volatilization ratio (%); m_0 is the weight of molybdenite concentrate, g; ε_0 is the Mo or Re weight fraction of molybdenite concentrate, %; m_2 is the weight of roasted product, g; and ε_2 is the weight fraction of Mo or Re of roasted product, %.

3. Results and Discussion

3.1. Removal of Pb by Acid Leaching with HCl

3.1.1. Chemistry of HCl Leaching

In HCl solution, galena is capable of being selectively removed due to its high solubility. Conversely, molybdenite remains chemically stable. Traditional processes of removing galena from molybdenite concentrate with HCl are carried out in the presence of CaCl₂ or NaCl [28]. Due to the existence of CaCO₃ (Table 1) in the raw concentrate, the newly formed CaCl₂ may facilitate the dissolution of PbCl₂. Thus, no additional CaCl₂ is needed during acid leaching. The chemical reactions can be described by the following equations:



During the acid leaching, calcite is easily dissolved (Equation (3)) and galena will react with hydrochloric acid to form lead chloride (Equation (4)). The lead chloride is soluble in hot water, and its solubility is improved distinctly in CaCl₂ solution by forming a complex compound (Equation (5)). As a result, the galena and calcite can be effectively removed and lead chloride is expected to be recovered by cooling the leachate [30].

3.1.2. Effect of HCl Concentration

The effect of hydrochloric acid concentration on the removal of Pb was investigated by keeping leaching temperature at 95 °C, leaching time of 10 min and liquid-solid ratio of 5 (mL/g). The results are plotted in Figure 3. It can be observed that lead was not effectively removed until the hydrochloric acid concentration was higher than 6 wt. %. By increasing the HCl concentration from 1 wt. % to

6 wt. %, the removal of Pb increased dramatically from 23.4% to 92.5%, and then it remained almost constant. During the acid leaching, hydrochloric acid is not only used to remove galena, but also exhausted by CaCO_3 , MgCO_3 as well as other sulfides. The HCl concentration required for leaching galena is actually a small part. The stoichiometric threshold of HCl concentration for removing the majority of Pb, Ca, Mg and part of Fe is about 1.5%–2%. As observed from Figure 3, the dramatic increase in Pb extraction is 2–3 times the stoichiometric threshold. Meanwhile, the leaching ratio of molybdenum increased gradually to its maximum value of 1.7% as the HCl concentration was increased to 6 wt. %. A further increase in HCl concentration lowers the leaching ratio of molybdenum. In contrast, the leaching ratio of rhenium kept increasing with HCl concentration. The suitable HCl concentration was 8 wt. %.

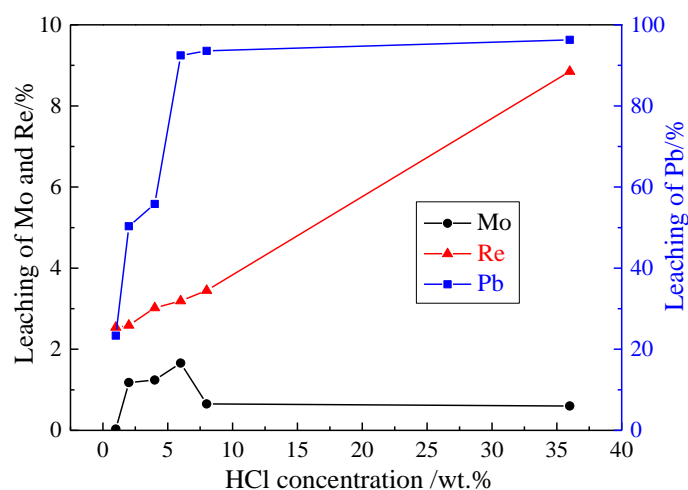


Figure 3. Effect of HCl concentration on the leaching of Mo, Re and Pb.

3.1.3. Effect of Leaching Temperature

By fixing the HCl concentration at 8 wt. %, leaching time at 10 min and liquid-solid ratio at 5 (mL/g), the effect of leaching temperature on the removal of lead and leaching of molybdenum and rhenium was examined, as shown in Figure 4. As the leaching temperature was increased, the reaction between galena and hydrochloric acid was accelerated and the solubility of PbCl_2 was improved. The leaching ratios of Pb and Re increased gradually from 24.5% and 0.5% to 93.6% and 3.6%, respectively, when the leaching temperature was increased from 25 °C to 95 °C. In addition, the little Mo was extracted in this regard, and the appropriate leaching temperature was 95 °C.

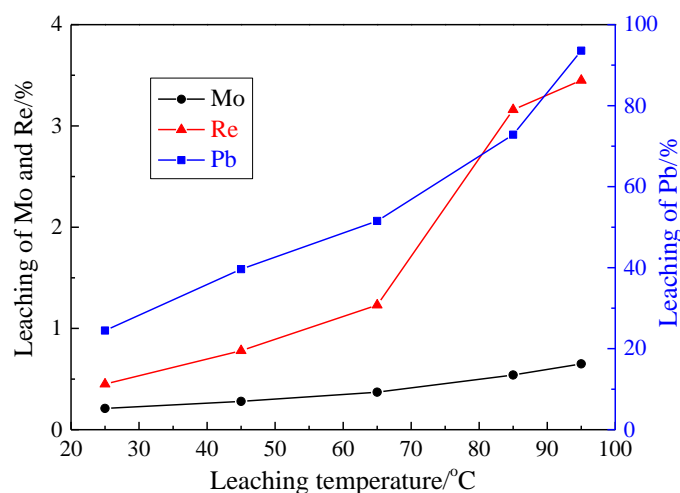


Figure 4. Effect of leaching temperature on the leaching of Mo, Re and Pb.

3.1.4. Effect of Leaching Time

The effect of leaching time on the removal of Pb was studied under the following conditions: HCl concentration of 8 wt. %, leaching temperature of 95 °C and liquid-solid ratio of 5 (mL/g). The results plotted in Figure 5 indicate that 85.5% of Pb was leached in only 2 min, and this number increased gradually to 93.6% in 10 min. The variation of Re was similar to that of Pb, which increased from 0.6% to 3.6% within the time range of 2–10 min. However, the leaching ratio of Mo changed slightly when the leaching time was prolonged to 60 min. Hence, leaching time of 10 min was sufficient for removing the majority of lead.

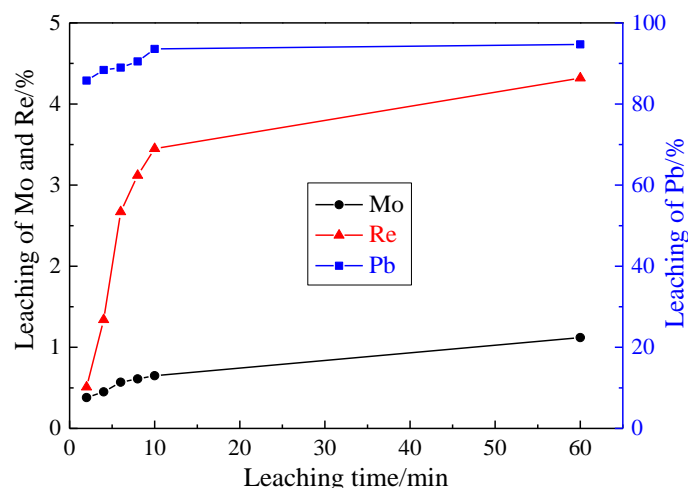


Figure 5. Effect of leaching time on the leaching of Mo, Re and Pb.

3.1.5. Effect of Liquid-Solid Ratio

Aiming at optimizing the liquid-solid ratio (mL/g) for complete removal of Pb, the experiments were performed with the liquid-solid ratio ranging from 2 to 6. The HCl concentration, leaching temperature and time were maintained at 8 wt. %, 95 °C and 10 min, respectively. From the results shown in Figure 6, it is ascertained that the removal of Pb increased significantly from 74.5% to more than 90% as the liquid-solid ratio was increased to 5. The leaching ratio of Re maintained at less than 4% when the liquid-solid ratio was smaller than 5 and increased distinctly thereafter. However, the leaching ratio of Mo decreased by increasing the liquid-solid ratio up to 5. Thus, the most suitable liquid-solid ratio was 5.

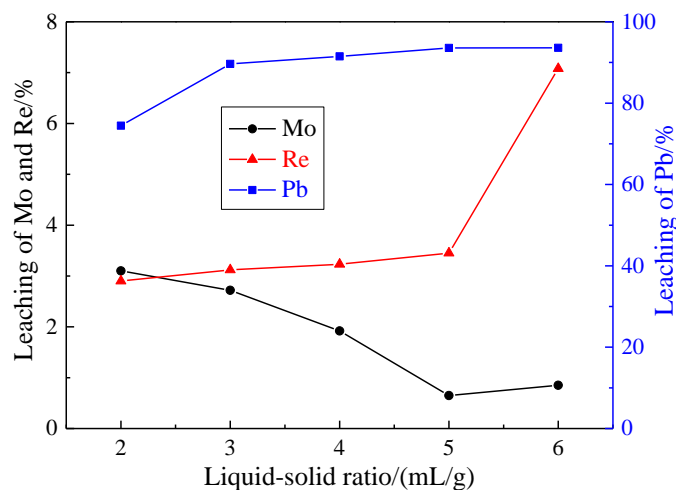


Figure 6. Effect of liquid-solid ratio on the leaching of Mo, Re and Pb.

From the above analysis, the suitable leaching conditions for removing Pb are as follows: HCl concentration of 8 wt. %, leaching temperature of 95 °C, leaching time of 10 min and liquid-solid ratio of 5. Most of galena (93.6%) and calcite (97.4%) were dissolved in the hydrochloric acid. The main chemical composition of molybdenite concentrate after the pretreatment is summarized in Table 2. It can be observed that Mo and Re were enriched after leaching due to the removal of galena and calcite.

Table 2. Main chemical composition of molybdenite concentrate after pretreatment.

Component	Mo	Re	Fe	SiO ₂	Al ₂ O ₃	CaO	MgO	WO ₃	Cu	Pb	S
wt. %	50.25	363 g/t	1.07	4.72	0.53	0.10	0.18	1.76	0.02	0.34	33.50

3.2. Separation of Rhenium by Oxidative Roasting

Oxidative roasting followed by ammonia leaching is the most classical process for recovering Mo and Re from molybdenite concentrate [22,23]. During the oxidative roasting, molybdenum disulfide is oxidized to molybdenum trioxide, which is soluble in the ammonia solution, while rhenium was volatilized into the flue dust in the form of Re₂O₇ (whose vapor pressure is 1 atm at 362 °C). In this section, oxidative roasting of the concentrate after acid leaching was conducted to investigate the effect of the pretreatment on the separation of rhenium.

3.2.1. Effect of Roasting Temperature

The effect of roasting temperature on the separation of Re is shown in Figure 7. Oxidative roasting was carried out for 120 min under the air atmosphere. The results in Figure 7 indicate that the volatilization of Re increased slowly and then remained almost constant at temperatures above 550 °C. Almost complete volatilization of rhenium was achieved for the concentrate after pretreatment, 10% higher than that without pretreatment. This is mainly attributed to the generation of lead molybdate and calcium molybdate in the presence of galena and calcite, which results in serious sintering and restrains the diffusion of O₂. Additionally, the formation of pererrhenate restricted the Re volatilization [25].

On the contrary, the volatilization of Mo before and after pretreatment gradually increased to 5.3% and 6.2%, respectively, when the roasting temperature was elevated to 600 °C. Moreover, a further increase in roasting temperature had a significant impact on the Mo volatilization. This is because molybdenum oxides formed during roasting would also partially sublime even below their melting points. Furthermore, higher temperature accelerated their sublimation. Considering the volatilization of Re, the roasting temperature could be decreased from 600 °C to 500–550 °C for the concentrate after pretreatment.

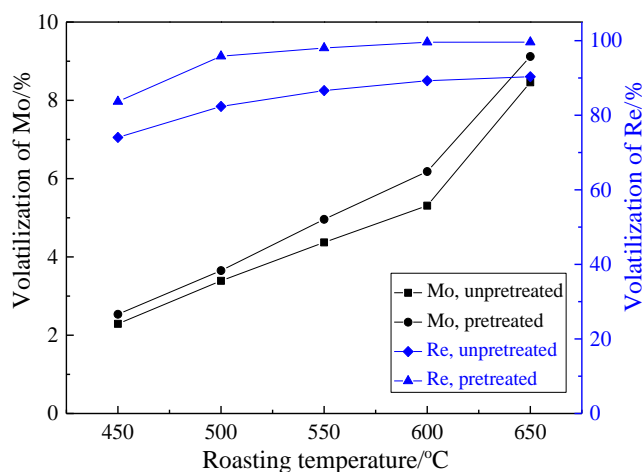


Figure 7. Effect of roasting temperature on the volatilization of Mo and Re.

3.2.2. Effect of Roasting Time

The effect of roasting time on the separation of Re was investigated and the results are shown in Figure 8. The pellets were roasted at 600 °C (unpretreated) or 550 °C (pretreated) in air for different time periods from 10 min to 150 min.

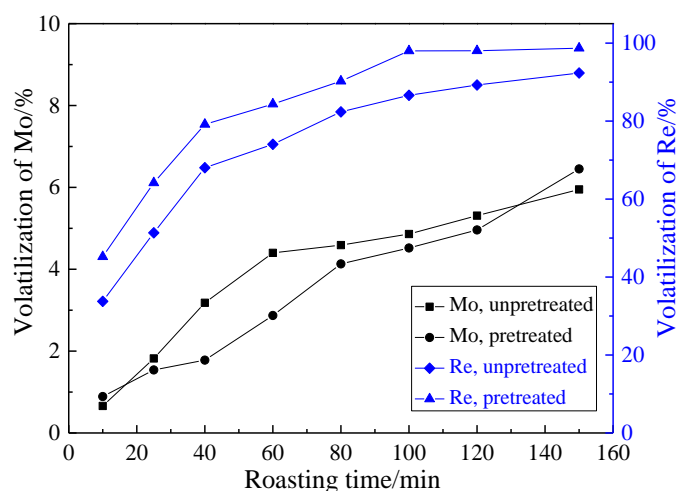


Figure 8. Effect of roasting time on the volatilization of Mo and Re.

It is found that the Mo volatilization increased steadily with increasing roasting time, and the volatilization ratio of Mo in the raw material was greater than that after pretreatment. This is mainly attributed to the higher requirement on the roasting temperature for the raw concentrate.

Similarly, the volatilization ratio of Re increased gradually with the increase of roasting time from 10 min to 100 min and then varied slightly. It is also evident that the rhenium volatilization was always higher than that without pretreatment under the experimental conditions. The maximum Re volatilization ratio reached 98.0% in 100 min, which was greater than that (86.6%) for the untreated concentrate. Thus, 100 min is adequate for roasting the concentrate after hydrochloric acid leaching.

3.2.3. Effect of O₂ Partial Pressure

The effect of O₂ partial pressure ($O_2/(O_2 + N_2)$) on the separation of rhenium from molybdenum is shown in Figure 9. The experiments were carried out under the conditions of 600 °C, 120 min (unpretreated) and 550 °C, 100 min (pretreated), respectively. The results in Figure 9 indicate that both the Mo and Re volatilization ratios of the raw concentrate reached their maximum values as the O₂ partial pressure was increased to 21%, and then decreased gradually. The sintering phenomenon becomes more serious under higher O₂ partial pressure, causing more compact structures of the roasted pellets [25].

After the molybdenite concentrate was pretreated by acid leaching, Mo volatilization kept increasing with increase in the O₂ partial pressure. In contrast, the volatilization of Re was improved and then remained nearly stable when the O₂ partial pressure exceeded 21%. The maximum Re volatilization ratio of 98.0% was obtained at 21% of O₂ partial pressure, while the corresponding Mo volatilization was only 4.5%. Thus, air atmosphere was recommended for oxidative roasting.

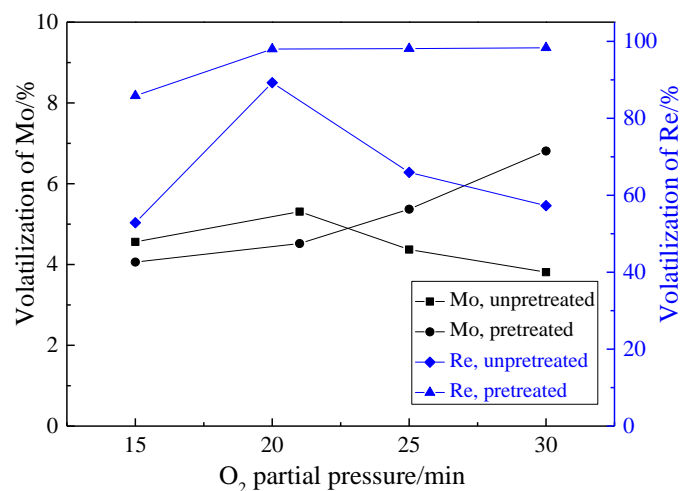


Figure 9. Effect of O₂ partial pressure on the volatilization of Mo and Re.

3.2.4. Phase Transformation in the Roasting Process

To identify the phase transformation of molybdenite concentrate during oxidative roasting, XRD analysis was performed for determination of the phase compositions of roasted products. The diffractograms of the raw material and roasted products are compared in Figure 10.

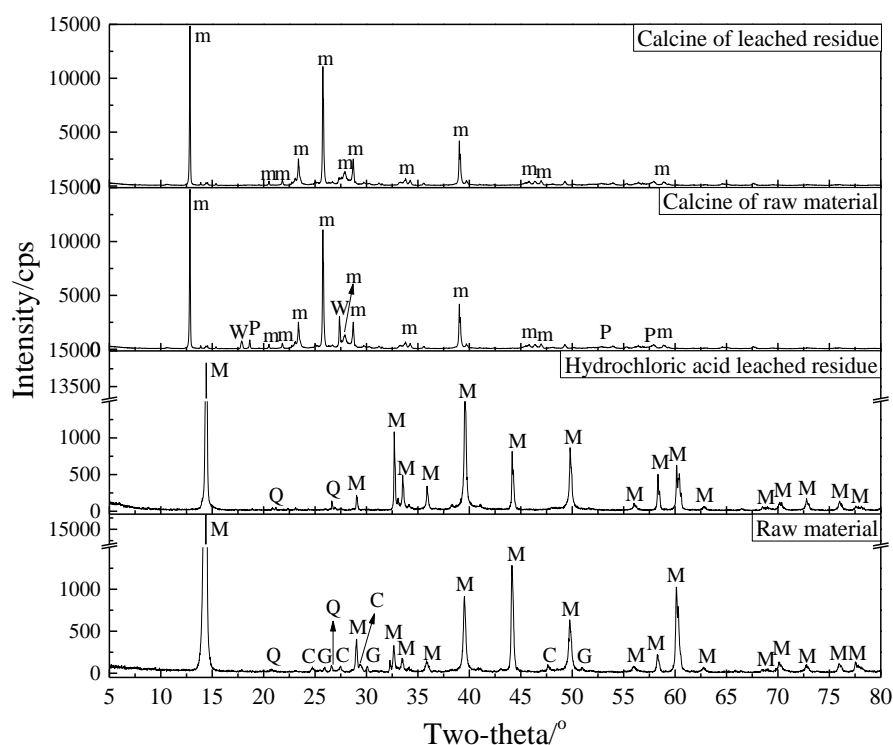
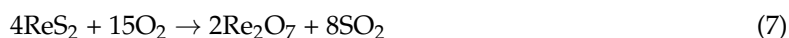


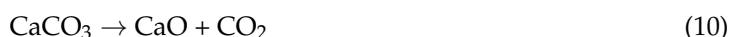
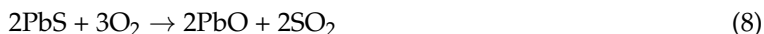
Figure 10. XRD patterns of the molybdenite concentrate and roasted products. (M—Molybdenite, MoS₂; G—Galena, PbS; C—Calcite, CaCO₃; Q—Quartz, SiO₂; m—Molybdite, MoO₃; P—Powellite, CaMoO₄; W—Wulfenite, PbMoO₄).

As observed in Figure 10, the diffraction patterns of roasted products are different from that of the raw molybdenite concentrate. Molybdenite (MoS₂) in the raw material disappeared, whereas molybdite (MoO₃) was formed according to the following equations [26,27]. Meanwhile, rheniite (ReS₂) was also oxidized to rhenium (VII) oxide (Re₂O₇) whose vapor pressure reached 1 atm at

362.4 °C. Thus, rhenium oxide is absent in Figure 10, while molybdate was retained in the calcine during oxidative roasting:



By roasting the raw concentrate at 600 °C for 120 min under air atmosphere, galena (PbS) was oxidized to lead oxide (PbO), which combined with molybdate to form wulfenite (PbMoO₄). In the meantime, calcite (CaCO₃) in the raw material was decomposed to calcium oxide (CaO). Similarly, CaO would also react with molybdate to generate powellite (CaMoO₄), as described by the following equations:



As a result of hydrochloric acid leaching, the peaks of galena and calcite were disappeared. No obvious diffraction peaks of wulfenite and powellite except those of molybdate are observed in the XRD pattern of the roasted product, which was obtained at 550 °C for 100 min under air atmosphere.

To evaluate the effect of removing lead and calcium on the leaching of Mo, the roasted products were leached at 60 °C for 3 h with 7 mol/L aqueous ammonium solution and liquid-solid ratio of 10 [4]. The results indicate that only 79.3% Mo was leached from the calcine of molybdenite concentrate without pretreatment. In contrast, the Mo leaching ratio reached 91.5% for the calcine of concentrate pretreated by acid leaching. This is attributed to the elimination of adverse effects of molybdates, which were insoluble in the aqueous ammonium solution. From the above results, it can be concluded that acid leaching as a method of molybdenite concentrate pretreatment not only played a significant role in improving the Re volatilization, but also had a crucial impact on the subsequent leaching of Mo.

4. Conclusions

Separation of rhenium from a kind of lead-rich molybdenite concentrate by oxidative roasting was studied. The volatilization of Re and leaching ratio of Mo in ammonia solution were improved significantly after the lead and calcium components in the raw material were removed by hydrochloric acid. The results of acid leaching show that 93.6% of Pb and 97.4% of Ca were removed under the conditions of 95 °C for 10 min with HCl concentration of 8 wt. % and liquid-solid ratio of 5 (mL/g). The oxidative roasting experiment indicates the rhenium volatilization of 89.3% was obtained for the raw concentrate when roasted at 600 °C for 120 min in air. In contrast, the rhenium volatilization was enhanced distinctly to 98.0% when the pretreated concentrate was roasted at 550 °C for 100 min. Furthermore, the molybdenum leaching ratio of 91.5% was achieved by leaching the calcine of pretreated concentrate in aqueous ammonia solution. Conversely, only 79.3% of Mo was leached from the calcine of molybdenite concentrate without pretreatment. The XRD analysis confirms that the formation of molybdates was adverse for the rhenium separation and the subsequent leaching of molybdenum, which can be prevented after acid pretreatment.

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Author Contributions: Guanghui Li and Zhiwei Peng conceived and designed and performed the experiments; Zhixiong You, Hu Sun and Rong Sun performed the experiments, analyzed the data and wrote the paper; Yuanbo Zhang and Tao Jiang supervised experimental work and reviewed the manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

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