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Leaching Behavior of the Main Metals from Copper Anode Slime during the Pretreatment Stage of the Kaldor Furnace Smelting Process

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Abstract: The Kaldor furnace smelting process is currently the mainstream process for treating copper anode slime, but the existence of copper, tellurium and other impurities has adverse effects on the recovery of gold and silver during the Kaldor furnace smelting stage. Therefore, it is necessary to pretreat the copper anode slime to remove these impurities before Kaldor furnace reduction smelting. However, the current pretreatment process of copper anode slime generally has the problem of low removal efficiency of copper and tellurium, and little research on the occurrence state of main metals in copper anode slime. Therefore, this study quantitatively determined the phase composition of Cu, Te, Pb, Bi, As, Sb, Se, Ag and Au, and hydrogen peroxide was introduced to enhance the leaching of impurities. The leaching behavior of each metal in copper anode slime was investigated in detail. The results demonstrate that Cu and Te in the copper anode slime mainly exist in the form of CuO and $CuSO_4$ and Te and $AuTe_2$, respectively. More than 99% of the Cu and 97% of the Te were leached out using 250 g/L H_2SO_4 and 28.8 g/L H_2O_2 with a leaching pressure of 0.8 MPa at 150 °C for 2 h, while the leaching of Au and Ag was both < 0.03%. The removal of Cu and Te and the enrichment of precious metals were achieved. This study provides a rich theoretical reference for the optimization of the Kaldor furnace process.

Keywords: copper anode slime; pressure acid leaching; leaching behavior; precious metals; Kaldor furnace smelting process

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1. Introduction

Rare and precious metal elements have excellent physical and chemical properties, such as high-temperature oxidation and corrosion resistance, electrical properties (excellent electrical conductivity, high-temperature thermoelectric properties and stable temperature coefficient of resistance, etc.), high catalytic activity, and strong coordination. These elements are widely used in the industry, so they are called the modern "industrial vitamin" [1–6]. However, the stock of rare and precious metals in the Earth's crust is scarce with the consumption of resources, and the cost and pollution to extract them directly are high because of their limited concentration in ores. Copper anode slime is an important raw material for the extraction of rare and precious metals. Copper anode slime is a byproduct of electrolytic copper refining. It is rich in large amounts of gold, silver, selenium, tellurium, antimony, bismuth, platinum groups and other rare and precious metals and contains lead, arsenic, cadmium, etc., which are potentially harmful to the environment [7]. According to statistics, approximately 50% of gold, 60% of silver, 70% of selenium, 90% of tellurium, 35% of platinum and 50% of palladium in China are produced from copper anode slime [8]. Usually, the yield of copper anode slime accounts for 0.2% to 0.8% [9] of the refined copper output. China is one of the largest copper producers in the world. The copper output of the mine in 2021 reached 1.8 million tons [10], and is expected to generate approximately

80,000 tons of copper anode slime, which places huge environmental pressure. At the same time, copper anode slime also provides great prospects for recycling valuable metals, and the economic value and environmental benefits are increasingly prominent.

At present, many scholars have conducted in-depth research on the treatment of copper anode slime, and there are four widely used methods: (1) The hydrometallurgical process, including the full-wet process and semiwet process, still suffers from complex equipment connections and unstable production quotas [11,12]. (2) The combined process of beneficiation and metallurgy has the advantages of less flue gas and a short production cycle but has the disadvantages of large wastewater discharge and low recovery of selenium [13,14]. (3) The traditional pyrometallurgical process is relatively mature but has the disadvantages of a long production cycle, high energy consumption and significant pollution [15–17]. (4) The Kaldor furnace process has the advantages of simple operation, strong adaptability of raw materials, less pollution, high gold and silver recovery and low energy consumption compared with the traditional pyrometallurgical process. The Kaldor furnace smelting process is an efficient anode slime treatment method. It operates in a continuous rotation condition, there is no dead space in the furnace body, and each part is fully stirred, so this process has a good kinetic condition. In addition, the use of the Kaldor furnace instead of the precious lead furnace and the silver separation furnace to complete the reduction smelting and oxidation blowing process will have positive impacts on reducing energy consumption, shortening the process flow, reducing environmental pollution and improving the operation environment [18]. For these reasons, many large copper companies, such as KGHM Polska Miedz and Polidon Ronnskar Smelter in Sweden, have gradually started to employ this production process.

Because the existence of copper and tellurium will produce copper matte in the Kaldor furnace reduction smelting process, copper matte can dissolve precious metals, reduce the direct yield of precious metals, and extend the time of the oxidation blowing. Therefore, the first step of comprehensive utilization of copper anode slime is to remove some base metals mainly composed of copper and tellurium through the chemical method; this process is called the pretreatment of copper anode slime. The quality of copper anode slime pretreatment process often determines the quality of the anode slime comprehensive recovery process. The copper removal methods are generally divided into three types: oxidation roasting-acid leaching, sulfation roasting-acid leaching, and direct acid leaching. Among them, the disadvantages of oxidation roasting-acid leaching and sulfation roasting-acid leaching are that smoke and dust production are during roasting and the working environment is poor. The direct acid leaching method (divided into normal pressure acid leaching and pressure acid leaching) uses sulfuric acid as the leaching agent and realizes copper removal under the condition of adding an oxidant. Compared with the above methods, the direct acid leaching method has the advantage of a good working environment [19].

The current method of copper anode slime pretreatment for copper removal is generally pressure acid leaching, that is, pressure acid leaching with oxygen as the oxidizing agent and sulfuric acid as the leaching agent. This approach is considered an alternative green enhanced leaching process. The advantages are cleanliness and environmental friendliness without the addition of other complex reagents [20]. Under the optimum conditions of pressure acid leaching, the leaching of Cu and Te can reach 97.71% and 59.62%, respectively [21]. Te can be recovered from copper telluride residues, and the recovery of tellurium can reach 93% with a Na_2SO_3/Te molar ratio of 3.2 at 85 °C for 2 h [22]. Elemental As can be removed from the copper anode slime by oxygen-enriched roasting followed by acid leaching. The volatilization rate of As was only 1.13% under the optimum oxygen-enriched roasting parameters. The results of acid leaching showed that the leaching efficiencies of As were above 97% [23]. Rao [24] used a hydrothermal oxidation method in a dilute H_2SO_4 solution to study the effect of the H_2SO_4 concentration on Cu and Se recovery. The transformation mechanism of selenide was clarified, and the optimal reagent concentration was achieved. The optimal H_2SO_4 concentration was selected as 0.5 mol/L, and above 99%

Cu and Se was extracted in a 0.5 mol/L H_2SO_4 solution with a liquid solid ratio of 10 mL/g and oxygen partial pressure of 1.0 MPa at 150 °C. To improve the recovery of gold from the copper anode slime, Zhang [25] reduced the gold chloride by controlling the potential. The relationship among the solution potential, concentration of gold ions, and reduction rate of gold were explored, and the results indicated that the concentration of gold ions can be reduced to <5 mg/L and the reduction rate of gold is >99.7% when the solution potential is <550 mV, showing the satisfactory reduction of gold chloride in solution. Sb and Bi are easier to leach in the Cl^- containing system. Researchers [26] found that the leaching of Sb and Bi reached 86% and 98% using 120 g/L H_2SO_4 and 140 g/L Cl^-, respectively, with an L/S of 4:1 at 80 °C for 3 h. However, the use of hydrochloric acid–sulfuric acid as a leaching agent, while allowing a large amount of bismuth and antimony into solution, also resulted in the partial leaching of silver and lead in the form of coordination compounds [27].

All the above studies only analyzed the leaching behavior of one or several elements in copper anode slime, and the leaching behavior of all major elements in the leaching process was not systematically studied. Therefore, in this study, based on the detailed analysis of the occurrence state of Cu, Te, Pb, Bi, As, Sb, Se, Ag and Au in copper anode slime, and improving the existing pressure leaching process, a high efficiency removal of Cu and Te is realized. This process provides a new idea for the healthy development of industrial copper anode slime.

2. Experiment

2.1. Experimental Materials

The experimental raw material is the copper anode slime from a smelter in northern China. The particle size of 90% of the copper anode slime is below 16.2 µm. The chemical compositions of the copper anode slime are presented in Table 1. The copper anode slime contains 4.85 wt% Ag, 0.58 wt% Au and 8.09 wt% Se, whereas the Cu, Pb, Bi and As contents are 19.33 wt%, 15.24 wt%, 14.18 wt% and 6.08 wt%, respectively, which demonstrates that this copper anode slime has considerable recovery value. From the X-ray diffraction pattern of the raw materials, as shown in Figure 1, it can be clearly observed that several diffraction peaks are related to CuS, $PbSO_4$, $BaSO_4$, Ag_2O, and Bi_2O_3.

Table 1. Main chemical components of copper anode slime.

Element	**Cu**	**Pb**	**Bi**	**Se**	**As**	**Ba**	**Ag**	**Te**	**Sb**	**Au**
Content (wt%)	19.33	15.24	14.18	8.09	6.08	5.39	4.85	1.76	0.98	0.58

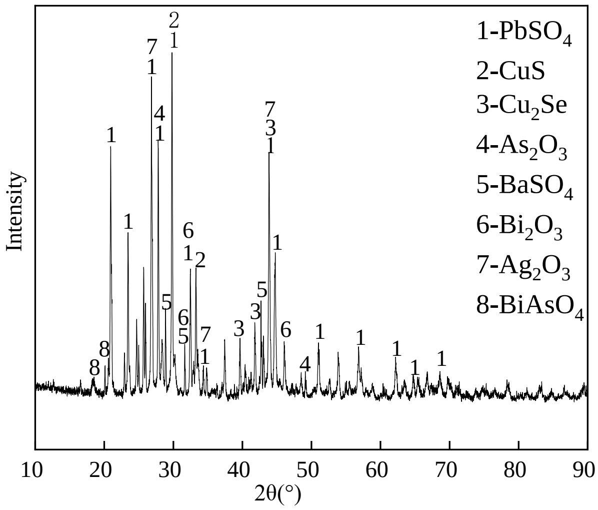

Figure 1. X-ray diffraction pattern of the copper anode slime.

Figure 2 illustrates the distribution maps of the main elements present in the coppered anode slime. Copper and tellurium were found to be uniformly distributed in the raw material. To further determine the metal occurrence state and content of copper anode slime, the main mineral phases of the raw material were identified by chemical analysis. The results are shown in Table 2. The results show that Cu is mainly present as CuO, $CuSO_4$, and CuS, Te is mainly present as Te and $AuTe_2$, and Pb, Bi and Ag are mainly present as $PbSO_4$, Bi_2O_3, and Ag_2S, respectively.

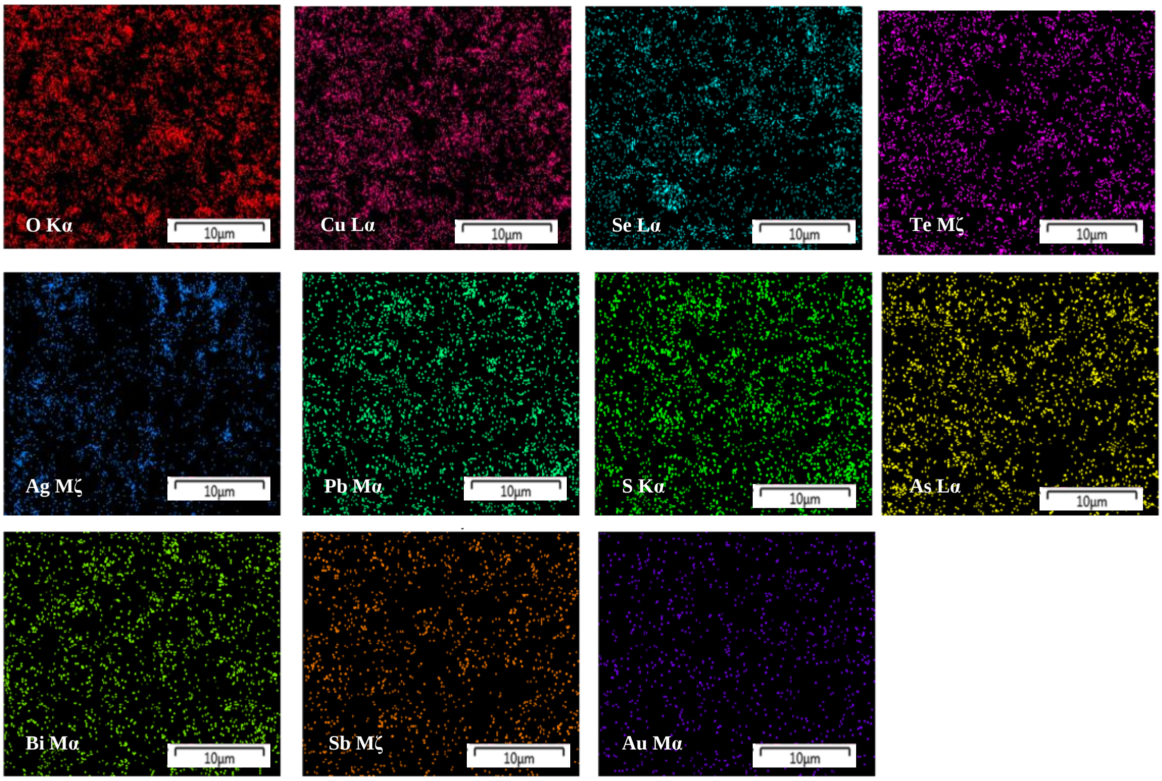

Figure 2. Distribution maps of the main elements present in the coppered anode slime.

Table 2. Mineral phase composition of copper anode slime.

Element	Phase	Content%
Cu	CuO	51.5
	$CuSO_4$	23.4
	CuS	21.0
	Cu	3.3
	$Cu_2(Se, Te)$	0.9
	$\sum Cu$	100
Se	Se	33.3
	Ag_2Se	39.3
	$SeO_2 + MeSeO_4$	1.9
	$PbSeO_4 + MeSeO_3$	13.6
	$Cu_2Se + PbSe$	10.9
	Au_2Se	1.0
	$\sum Se$	100
Ag	Ag	2.03
	Ag_2O	2.34
	$AgSO_4$	0.13
	Ag_2SAg_2Se	53.9235.17
	Ag_2Te	6.41
	$\sum Ag$	100

Table 2. *Cont.*

Element	Phase	Content%
Te	Ag_2Te	6.1
	CuTe	9.6
	Te + $AuTe_2$	84.3
	∑Te	100
Bi	Bi	17.0
	Bi_2S_3	9.2
	Bi_2O_3	73.9
	∑Bi	100
Au	Au	65.39
	Au_2Se	5.86
	$AuTe_2$	28.76
	∑Au	100
Sb	Sb	7.1
	Sb_2O_3	72.8
	Sb_2S_3	1.5
	Metal solid solution	18.6
	∑Sb	100
As	As_2O_3	1.1
	As_2S_3	8.6
	$MeAsO_4$	87.7
	Residue	2.7
	∑As	100
Pb	Pb	12.35
	PbO	3.70
	$PbSO_4$	79.26
	PbS	4.69
	∑Pb	100

2.2. Experimental Principle and Method

In the process of pressure acid leaching of copper anode slime, Cu mainly enters the solution in the form of $CuSO_4$, Te will react with H_2SO_4 to form H_2TeO_3 or H_2TeO_4 into the solution, and rare and precious metals such as gold and silver remain in the leach residues to separate noble and base metals. The main reactions are

$$CuO + H_2SO_4 = CuSO_4 + H_2O \tag{1}$$

$$Cu_2Te + 2O_2 + 2H_2SO_4 = 2CuSO_4 + H_2TeO_3 + H_2O \tag{2}$$

$$Ag_2Te + 3O_2 + H_2SO_4 = Ag_2SO_4 + 2H_2TeO_3 \tag{3}$$

$$2Au_2Te + O_2 + 4H_2SO_4 = 4Au + 2H_2TeO_4 + 4SO_2 + 2H_2O \tag{4}$$

$$Cu_2Se + 2H_2SO_4 + 2O_2 = 2CuSO_4 + H_2SeO_3 + H_2O \tag{5}$$

$$2CuS + 2H_2SO_4 + O_2 = 2CuSO4 + 2H_2O + 2S \tag{6}$$

When the acidity in the system is high, sulfur will be oxidized by oxygen to form sulfuric acid, and the reaction is as follows:

$$2S + 3O_2 + 2H_2O = 2H_2SO_4 \tag{7}$$

The specified concentration of sulfuric acid is added to the beaker, a certain mass of copper anode mud is added and stirred well, and this mixture is transferred to the autoclave (ZNCL-DT4, from TaiKang, Technology Ltd., Xi'an, China) where it is pressurized with oxygen, and the temperature is set, with stirring at 300 rpm and a L/S ratio of 10:1. After leaching is completed, heating stops, the autoclave naturally cools to 30 °C, the pressure is released, the reactor liner is removed, and the reaction solution is extracted.

2.3. Analytical Method

The main phases of the samples are identified by X-ray diffraction (XRD; PANalytical X'Pert Pro Powder, Almelo, the Netherlands) using a CuKα radiation source with a 40 kV acceleration potential and current of 40 mA. XRD diffractograms are analyzed by using Jade 6.5 software. The concentrations of Cu, Pb, Se, Bi, Ag, Au, Sb, Te, and As are analyzed by inductively coupled plasma optical emission spectroscopy (ICP–OES, Perkin Elmer Optima 7100DV, Waltham, MA, USA). The morphology and element distribution behaviors in the copper anode slime are determined by SEM–EDS (MIRA 3 LMH, TESCAN Brno, S.r.o, Brno, Czech Republic). The chemical composition of residues is analyzed by X-ray fluorescence spectrometry (Philips PW2400).

3. Experimental Results and Discussion

3.1. Effect of the Leaching Time

The effect of the leaching time on the recovery of metals during the leaching stage is presented in Figure 3. It can be observed that the leaching of most elements increased with increasing leaching time. When the leaching time was extended from 0.5 h to 2.0 h, the leaching of Cu, Te and Se ranged from 72.02%, 38.03% and 13.08% to 85.10%, 56.49% and 26.29%, respectively. At 2 h, the leaching of Au, Ag, Pb and Bi were 0.04%, 0.03%, 0.021% and 0.025%, respectively, with almost no leaching. According to Table 2, bismuth in copper anode mud mainly exists in the form of Bi_2O_3. Bi_2O_3 reacts in the sulfuric acid system to form an insoluble substance, resulting in very low bismuth leaching, with the following reaction equation [28]:

$$Bi_2O_3 + 2H_2SO_4 = 2Bi(OH)SO_4\downarrow + H_2O \quad (8)$$

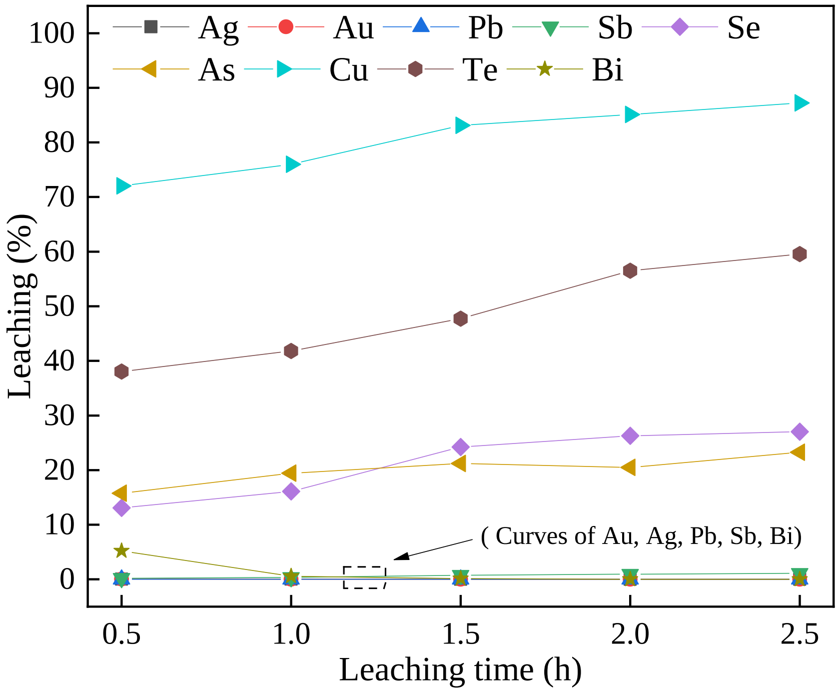

Figure 3. Effect of time on the leaching of each element (T = 150 °C, P = 0.8 MPa, H_2SO_4 = 100 g/L).

The XRD analyses of the leach residues are shown in Figure 4, from which it can be observed that the physical phases in the leach residues are mainly $BaSO_4$, $PbSO_4$, $BiAsO_4$, and Ag_3AsS_3, in which $BiAsO_4$ is the insoluble material produced by the coprecipitation reaction between As and Bi during the electrolysis of copper and cannot be dissolved in sulfuric acid [29].

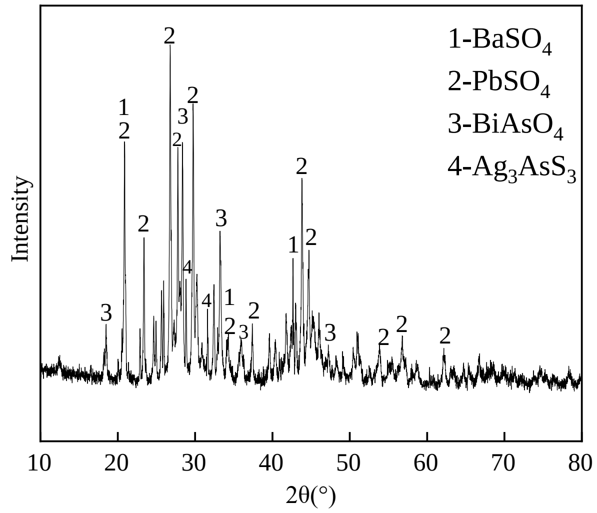

Figure 4. X-ray diffraction pattern of the leach residues (t = 2 h, T = 150 °C, P = 0.8 MPa, H_2SO_4 = 100 g/L).

3.2. *Effect of the Sulfuric Acid Concentration*

The effect of the H_2SO_4 concentration was examined by varying the H_2SO_4 concentration from 100 g/L to 300 g/L, as shown in Figure 5. The results indicate that the extraction of Cu, Te and As increases substantially as the H_2SO_4 concentration increases from 100 g/L to 300 g/L, with a change from 85.10%, 56.49% and 20.47% to 98.55%, 81.82% and 41.71%, respectively. At a H_2SO_4 concentration of 300 g/L, the leaching of Au, Ag, Pb and Sb were 0.006%, 3.81%, 0.10% and 0.93%, respectively. Ag in copper anode slime exists mainly in the form of Ag_2Se, Ag_2Te, and Ag_2S, all of which generate silver sulfate precipitates in the sulfuric acid system. The leaching of silver increases with increasing sulfuric acid concentration because the excess sulfuric acid continues to react with the generated silver sulfate to form $AgHSO_4$ [30], which is a soluble substance and has the following reaction equation:

$$Ag_2SO_4 + H_2SO_4 = 2AgHSO_4 \tag{9}$$

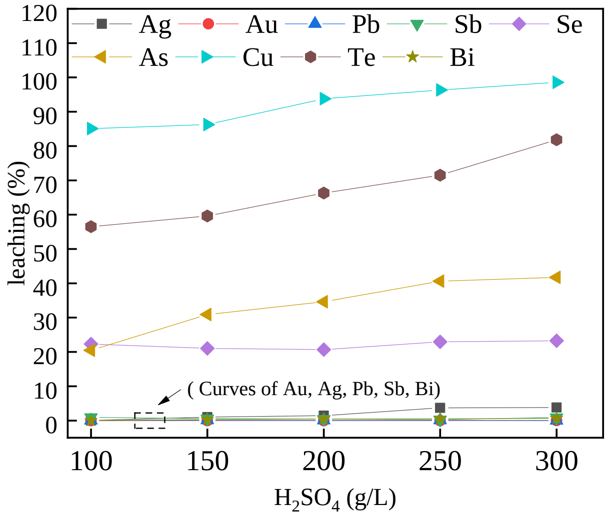

Figure 5. Effect of sulfuric acid concentration on leaching of each element (T = 150 °C, P = 0.8 MPa and t = 2 h).

The Cu in the raw material is mainly composed of copper oxide (51.5%), copper sulfate (23.4%), copper sulfide (21%) and a small amount of monomeric copper and copper selenide and telluride. Among them, copper oxide and copper sulfate can be easily leached in a sulfuric acid system, and monomeric copper and copper sulfide can be leached into solution under a strong oxidizing atmosphere. Te is mainly Te + $AuTe_2$ dominated and includes some Ag_2Te and CuTe, which are easily leached into the solution under strong acid and strong oxidizing properties, so the leaching of both copper and tellurium is high.

3.3. Effect of the Leaching Temperature

Figure 6 shows the effect of the temperature on the leaching of Cu, As, Bi, Ag, Au, Te, Sb, and Se. These results indicate that the leaching of Cu and Te increased from 92.84% and 67.54% to 97.89% and 72.37%, respectively, when the temperature was increased from 130 °C to 160 °C. Increasing the temperature can promote the decomposition of oxygen into oxygen atoms but can also reduce the solubility of oxygen. The leaching of Se increased from 16.09% to 22.9% when the leaching temperature increased from 130 °C to 150 °C. However, when the leaching temperature increased from 150 °C to 160 °C, the leaching of Se decreased to 20.5%. The cause of this phenomenon was the oxidation of selenium to selenium dioxide gas as the temperature increased [31]. The leaching of gold, antimony, Pb and Bi was less than 1% in the range of leaching temperatures from 130 °C to 160 °C. There was a slight increase in the leaching of Ag from 1.02% to 3.71% when the temperature increased from 150 °C to 160 °C. To reduce the leaching of silver, the temperature should not be too high.

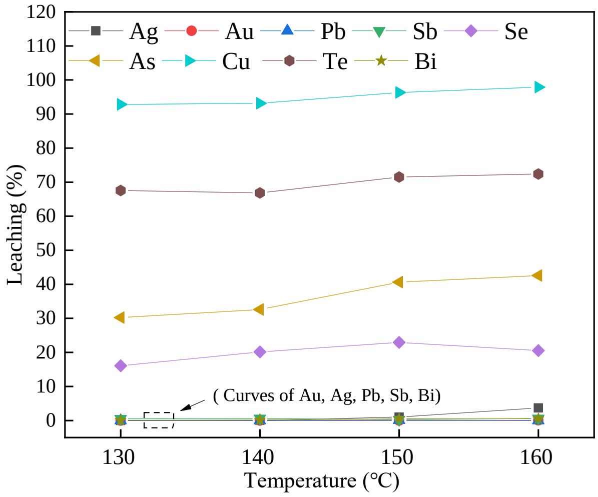

Figure 6. Effect of the temperature on the leaching of each element (t = 2 h, P = 0.8 MPa and H_2SO_4 = 250 g/L).

3.4. Effect of the Leaching Pressure

The yields of Cu, Pb, Se, Bi, Ag, Au, Sb, Te and As in the leach solution were investigated over a pressure range between 0.7~1.0 MPa, and the results are shown in Figure 7. With increasing pressure, the leaching of Cu, Te, As and Se increased. When the leaching pressure increased from 0.7 MPa to 1.0 MPa, the leaching of Cu and Se increased from 93.24% and 20.31% to 97.63% and 29.12%, respectively. The leaching of Te increased from 70.53% to 84.42%, and the leaching of Ag increased from 0.008% to 1.55%. To reduce the leaching of Ag, the leaching pressure should be reduced. Increasing the reaction pressure accelerated the rate of oxygen diffusion from the gas phase to the liquid phase, promoting the reaction. The leaching of Au, Pb and Bi was less than 1%. Pb mainly exists in the form of $PbSO_4$ (accounting for 79.26%) in copper anode slime, and the rest is composed of monomeric lead, lead oxide, and lead sulfide, which generate lead sulfate precipitation in the sulfuric acid system, resulting in a very low lead leaching. Au mainly exists in monomeric form (accounting for 65.39%), and this form of Au does not dissolve in the sulfuric acid system. The remaining gold is mainly Au_2Se and $AuTe_2$; this form of gold produces gold monomers in the process of pressure acid leaching, so gold is rarely immersed in solution.

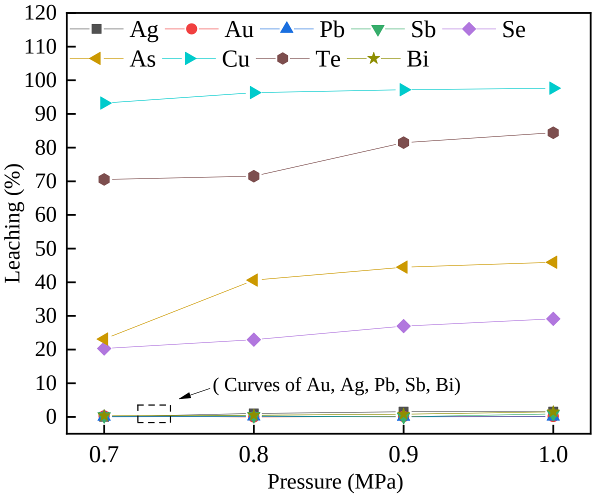

Figure 7. Effect of the leaching pressure on the leaching of each element (t = 2 h, T = 150 °C and H_2SO_4 = 250 g/L).

3.5. Effect of the Hydrogen Peroxide Concentration

The effect of the hydrogen peroxide concentration on the leaching behavior of Cu, Au, Ag, As, Bi and Sb are presented in Figure 8. These results suggest that the hydrogen peroxide concentration can have a significant effect on the leaching of copper and tellurium. The leaching of Cu and Te increased from 87.15% and 82.36% to 99.78% and 97.46%, respectively, when the hydrogen peroxide concentration increased from 7.4 g/L to 28.8 g/L. A further increase in the hydrogen peroxide concentration from 28.8 to 69.3 g/L resulted in only minor changes in the leaching of Cu and Te. There was a slight decrease in the leaching of Sb, from 43.28% to 38.64%, when the hydrogen peroxide concentration increased from 7.4 g/L to 69.3 g/L, because the addition of hydrogen peroxide oxidized antimony to high-valent oxides, and high-valent antimony oxide is difficult to dissolve in the sulfuric acid system [32]. The leaching of Ag, Au, and Pb were 1.20%, 0.01%, and 0.03%, respectively, with almost no leaching.

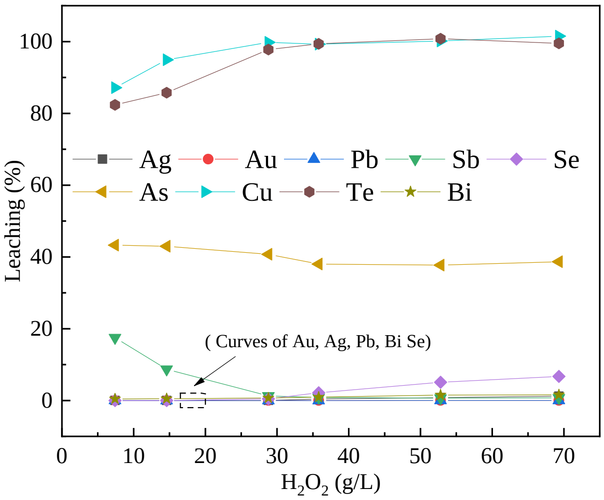

Figure 8. Effect of the hydrogen peroxide concentration on the leaching of each element (t = 2 h, T = 150 °C, P = 0.8 MPa and H_2SO_4 = 250 g/L).

The main element contents of the leach residue are shown in Table 3. The leach residue contained 18.06 wt% Pb, 14.66 wt% Bi, and 9.87% wt% Se, whereas the Ag, Au and As contents were 5.89 wt%, 0.69 wt% and 4.34 wt%, respectively. The tellurium content was only 0.33, and no copper was detected. The main mineral phases of the residue were identified by XRD. As shown in Figure 9, the main mineral phases present in the leach residue were $PbSO_4$, $BaSO_4$, $BiAsS_3$, and $BiAsO_4$.

Table 3. Main chemical composition of H_2O_2 = 28.8 g/L leach residue.

Element	Pb	Bi	Se	As	Ba	Ag	Sb	Au	Te
Content (wt%)	18.06	14.66	9.87	4.34	7.09	5.89	1.25	0.69	0.33

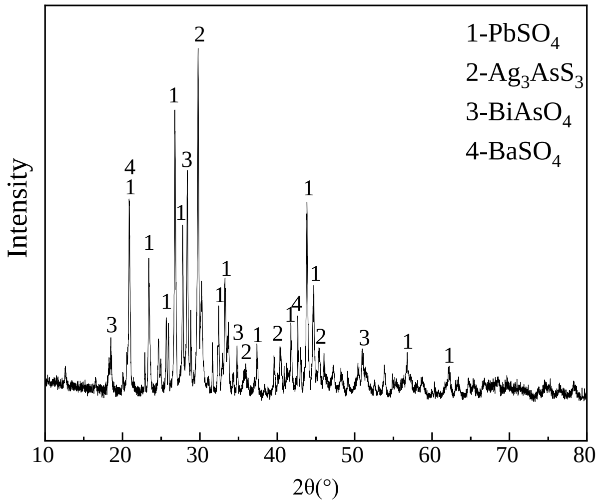

Figure 9. X-ray diffraction pattern of the leach residues (H_2O_2 = 28.8 g/L, t = 2 h, T = 150 °C, P = 0.8 MPa and H_2SO_4 = 250 g/L).

After extensive experimental investigations, 99.78% of Cu and 97.46% of Te were leached out using 250 g/L H_2SO_4 and 28.8 g/L H_2O_2 with a leaching pressure of 0.8 MPa at 150 °C for 2 h, indicating that Cu and Te were almost completely removed. Furthermore, the leaching rates of Au, Ag, Pb, Sb, Se, Bi and As were 0.02%, 0.0052%, 0.021%, 1.28%, 0.44%, 0.70% and 40.73%, respectively.

4. Conclusions

(1) More than 99% of Cu and 96% of Te were leached out using 250 g/L H_2SO_4 and 28.8 g/L H_2O_2 with a leaching pressure of 0.8 MPa at 150 °C for 2 h, and almost no leaching of gold and silver occurred. The leach residue was mainly composed of $BaSO_4$, $PbSO_4$, $BiAsO_4$, and Ag_3AsS_3. The separation of Cu and Te and the enrichment of precious metals were achieved.

(2) Cu in copper anode slime mainly exists in the form of CuO and $CuSO_4$, Pb mainly exists in the form of $PbSO_4$, Bi mainly exists in the form of Bi_2O_3, As mainly exists in the form of $MeAsO_4$, Te mainly exists in the form of Te and $AuTe_2$, and Ag mainly exist in the form of Ag_2S and Ag_2Se.

(3) In the sulfuric acid system, the leaching of gold, silver and bismuth is extremely low. Increasing the concentration of sulfuric acid or adding hydrogen peroxide can greatly improve the leaching of copper and tellurium. Increasing the leaching temperature and pressure has a greater effect on the leaching of arsenic but has less effect on the leaching of copper and tellurium.

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