

# Vacuum Gasification-Directional Condensation for Separation of Tellurium from Lead Anode Slime

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**Abstract:** Tellurium is the indispensable base material of semiconductors in solar panels. Traditional tellurium recycling, a highly complicated separation process, has exhausted reagents and energy sources whilst producing waste residue and water containing multitudinous heavy metal that is hugely harmful to the ecological environment. A clean and eco-friendly vacuum distillation-directional condensation treatment was investigated for its potential to recycle tellurium from tellurium-rich lead anode slime (TLAS). The optimal distillation temperature and response time conditions of 1173 K and 50 min were obtained based on a large number of experiments. Gasification results indicated that under the optimal conditions of distillation temperature 1173 K, constant temperature time 50 min, and system pressure 5–15 Pa, 92% of tellurium was volatilized and enriched into the condenser from TLAS. Condensate results revealed that 88% of elemental tellurium was directly recovered in the volatile matter. The appropriate gasification-condensation processes realized a clean utilization to extract tellurium and separate multi valuable metals effectively.

**Keywords:** lead anode slime; tellurium; gasification; directional condensation; clean recovery



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## 1. Introduction

Tellurium in the Earth's crust is small and dispersed, and the abundance of tellurium is  $6 \times 10^{-8}$ . Globally, the reserve of tellurium resources was 31 thousand tons, and the share of China accounted for 21% [1]. Tellurium is a rare metalloid, horned as a “vitamin of modern industry, national defense and cutting-edge technology” and “supporting materials for contemporary high-tech advanced materials” [2,3]. Tellurium has a vital role in metallurgy, chemical industry, electronics, etc. Tellurium is added to copper, lead, and iron alloys to improve their processing performance. Tellurium addition can improve copper alloy's cutting performance. Tellurium addition in lead can be used to fabricate cable jackets. Additionally, 0.03–0.04% of tellurium in steel can enhance steel's strong corrosion resistance [4]. The main applications of tellurium are tellurium and tellurium compounds as catalysts and additives in the chemical industry. Tellurium acts as a dispersant to improve rubber's strength and elasticity in rubber's industrial application [5]. Tellurium is a semiconductor material commonly used in electronic equipment. For example, mercury cadmium telluride is the material for making infrared-sensitive semiconductor materials. Cadmium telluride (CdTe) is the primary material for making solar panels. Tellurium can also be used to make gas sensors, photovoltaic modules, and phase change memory chips [6–9]. In addition, tellurium and tellurium compounds can be applied to treat bacterial infectious diseases, inhibit T cells (thymus-dependent lymphocytes) from cytokine

production, immunomodulatory activity and resistance to pathogens, and in vivo imaging and labeling cells in life science [10]. It follows that tellurium is a pivotal material in many fields, and the opinions on extracting tellurium are significant.

Currently, approximately 90% of tellurium was extracted from copper anode slime produced by crude copper's electrolytic refining. The remaining 10% of tellurium was recycled from the smelting and refining by-products of lead, bismuth, and nickel [2–4]. Methods for extracting tellurium from anode slime included sulfate roasting-electrolysis, sulfate roasting-reduction, sodium carbonate smelting, oxidation roasting-alkali leaching, chlorination-extraction, etc. Sulfuric acid roasting-electrolysis utilized the concentrated sulfuric acid roasting-soda smelting-soda oxidation refining-caustic soda electrolysis to obtain tellurium from the different anode slime. Sulfate roasting-reduction added  $\text{Na}_2\text{SO}_4$  for roasting and then added  $\text{Na}_2\text{SO}_3$  for reduction to separate tellurium. Oxidative roasting-alkali leaching utilized low-temperature roasting-NaOH leaching-electrolysis to obtain metal tellurium [11]. In some situations, these traditional methods both have different advantages, but the complicated flow exhausted chemical reagents and high-quality energy, and the roasted residue, the leached liquor, and the escaped gas become a more complex problem. Furthermore, tellurium will distribute in all process links from the widely used sulfation roasting electrolysis of copper and lead anode slime. The recovery rate of tellurium is only 75% [12]. Therefore, it is necessary to explore a clean, efficient, and environmentally friendly metallurgical method to extract tellurium from anode slime.

Vacuum metallurgy is a metallurgical process for smelting and refining metals and alloys in a closed environment below atmospheric pressure [13–15]. This method was widely used for secondary resource recovery, alloy separation, metal thermal reduction, and metal purification [16–19]. Vacuum metallurgy technology could be used to enrich and purify gold and silver from lead anode slime, separate lead–antimony alloy, lead–silver alloy, and bismuth–silver alloy, and deeply purify crude zinc and crude lead [20–22]. Compared with traditional smelting methods, vacuum metallurgical technology has some advantages: efficient resource utilization, high metal recovery rate, short metallurgical process, low energy consumption, good working conditions, and considerable economic benefits. It does not generate pollutants, such as waste gas, waste water, waste residue, and other contaminants [23–26]. This study, therefore, explored a novel vacuum method to recycle tellurium from TLAS, mainly adopting: (1) elucidate the element composition and occurrence state of TLAS, distilled residue, and volatile; (2) analyze the separation and recovery effects in terms of each pure substance and oxide's saturated vapor pressure in TLAS; (3) explore the distribution rule of tellurium in the vacuum gasification-directional condensation process. Finally, this work attempts to understand tellurium vacuum behavior and its further effect on the environment.

## 2. Experimental and Analysis

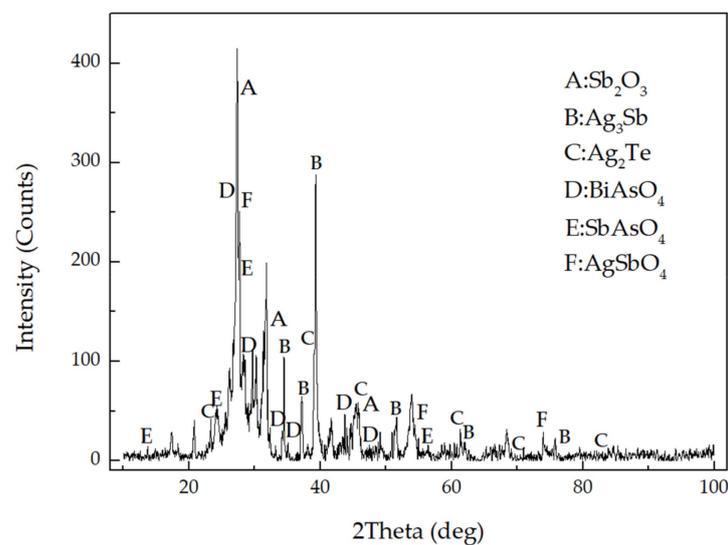
### 2.1. Experimental Materials

The raw material was an extraordinary TLAS produced by crude lead electrolysis in a lead smelter in Jiyuan city, Henan Province, China. The chemical compositions of TLAS were determined by atomic absorption spectrometry (AAS, WFX-320, Beijing Beifen-Ruili Analytical Instrument (Group) Corporation, Beijing, China), chemical titration, and inductively coupled plasma optical emission spectrometer (ICP-OES, OPTIMA 8000, PerkinElmer Corporation, Hong Kong, China). The chemical results (Table 1) show ten times more tellurium than located in another lead smelter's anode slime. The tellurium content reaches 0.21% (Table 1).

**Table 1.** Chemical compositions of TLAS (tellurium-rich lead anode slime) (wt%).

Component	As	Ag	Te	Bi	Pb	Sb	Cu	Au
wt/%	2.4	9.2	0.21	16	15	38	2.2	670 (g/t)

Solid samples were crushed and sieved before X-ray diffraction (XRD) analysis performed on a Rigaku/D-Mac-3c X-ray diffractometer (XRD, D-MAX/2600, Rigaku, Japan), with Cu K $\alpha$  radiation. XRD patterns were collected in 2 theta angle range from 10° to 90° at a scanning rate of 0.02°/s. XRD data used the Jade 6.5 to identify the crystal phase of the initial TLAS, the distilled volatile, and residue. The phases of TLAS acquired by XRD data were intermetallic compounds, oxides, and multiple oxides, in which Ag<sub>2</sub>Te was one of the occurrence forms of tellurium (Figure 1).



**Figure 1.** XRD (X-ray diffraction) pattern of TLAS (tellurium-rich lead anode slime).

## 2.2. Theoretical Analysis

### 2.2.1. Criterion of Saturated Vapor Pressure of Pure Substance

A substance's condensed state (solid or liquid) and its gaseous state will form a dynamic balance under the confined conditions of absolute pressure and specific temperature. The number of molecules converted from gaseous state to condensed state is equal to that of molecules converted from condensed state to gaseous state in unit time. This pressure is called the saturated vapor pressure of this substance at this temperature (referred to as vapor pressure). The same substance has different saturation autoclaves at different temperatures whilst increasing with temperature. The saturated vapor pressure of a pure solvent is greater than the saturated vapor pressure. The saturated vapor pressure of a solid in the same substance is less than the liquid's saturated vapor pressure. The greater the saturated vapor pressure of a substance at the same temperature, the easier it volatilizes. Comparing with saturated vapor pressure, the volatilization order of different substances can be determined. The relationship between saturated vapor pressure and temperature of a pure substance was expressed as Equation (1) [27]:

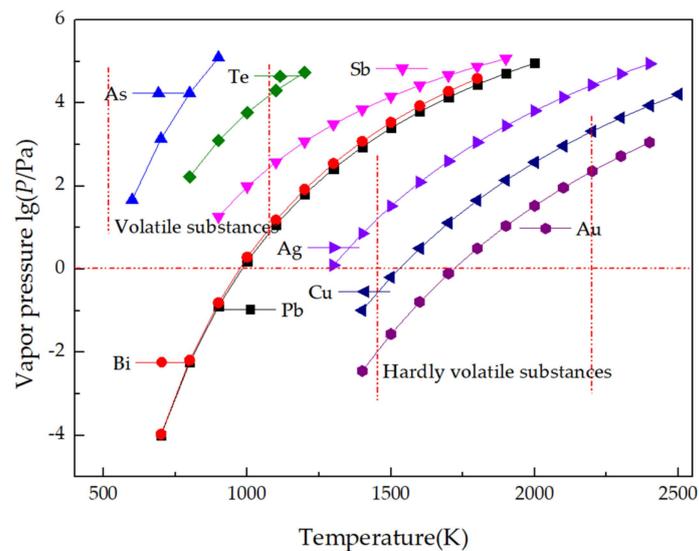
$$\lg p = AT^{-1} + B \lg T + CT + D \quad (1)$$

$A$ ,  $B$ ,  $C$ , and  $D$  are evaporation constants of each element. Different elements have different constants;  $p$  is the saturated vapor pressure in Pa;  $T$  is the temperature in K. The evaporation constants of components in TLAS were shown in Table 2.

**Table 2.** Evaporation constants of each component in TLAS.

Element	Pb	Bi	As	Sb	Te	Cu	Ag	Au
A	−10,130	−10,400	−6160	−6500	−7830	−17,520	−14,400	−19,280
B	−0.985	−1.26	0	0	−4.27	−1.21	−0.85	−1.01
$C \times 10^3$	0	0	0	0	0	0	0	0
D	13.28	14.47	11.94	8.49	24.41	15.33	13.82	14.50

Relationship results between temperature and saturated vapor pressure of each component in TLAS presented in Figure 2 show that tellurium, zinc, arsenic, antimony, and bismuth have a higher saturated vapor pressure. On the contrary, gold and silver have lower saturated vapor pressures at the same range of 500 to 2500 K. In addition, the saturated vapor pressure of tellurium is lower than that of arsenic at the same temperature, but is higher than that of antimony, bismuth, lead, etc. Therefore, arsenic will be preferentially volatilized into the gas phase. Antimony, bismuth, and lead will be volatilized immediately after tellurium gasification, which can be further cooled to a solid phase under vacuum cooling action. Precious metals of gold and silver will be non-volatilized and remained in the residues to achieve separation of tellurium.



**Figure 2.** Relationship between the saturated vapor pressure and temperature of each pure substance in TLAS.

### 2.2.2. Saturated Vapor Pressure of Oxides in TLAS

Metal oxides stably presented are lead oxide ( $PbO$ ), arsenic trioxide ( $As_2O_3$ ), antimony trioxide ( $Sb_2O_3$ ), tellurium dioxide ( $TeO_2$ ), and bismuth trioxide ( $Bi_2O_3$ ). Calculation of saturated vapor pressure of each oxide could be deduced as follows in terms of previous research [27]:

$$\lg p_{PbO} = -13,480T^{-1} - 0.92\lg T - 0.35 \times 10^{-3}T + 16.48 \quad (2)$$

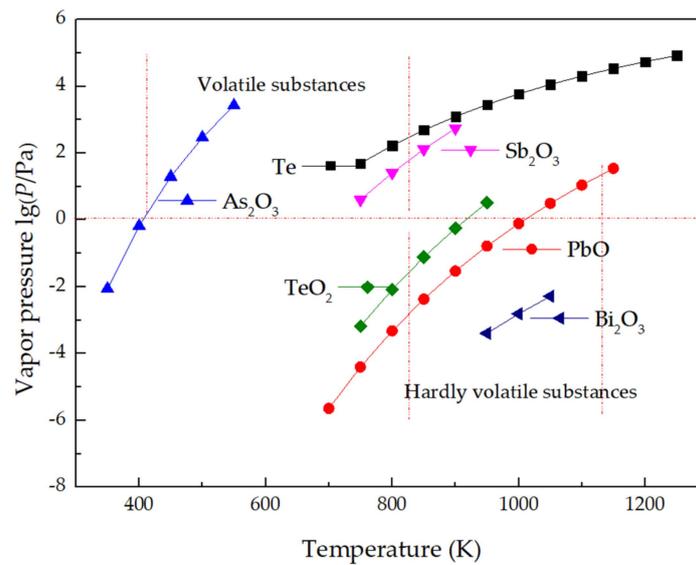
$$\lg p_{As_2O_3} = -5282T^{-1} + 13.03 \quad (3)$$

$$\lg p_{Sb_2O_3} = -9625T^{-1} + 13.44 \quad (4)$$

$$\lg p_{TeO_2} = -13,169T^{-1} + 14.38 \quad (5)$$

$$\lg p_{Bi_2O_3} = -10,150T^{-1} + 8.24 \quad (6)$$

Calculation results (Figure 3) of the relationship between each oxide's saturated vapor pressure in TLAS and temperature can be obtained by Equations (2)–(6).

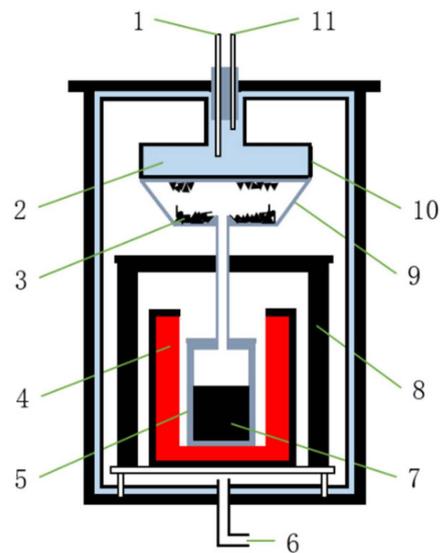


**Figure 3.** Relationship between the saturated vapor pressure and temperature of each metal oxide.

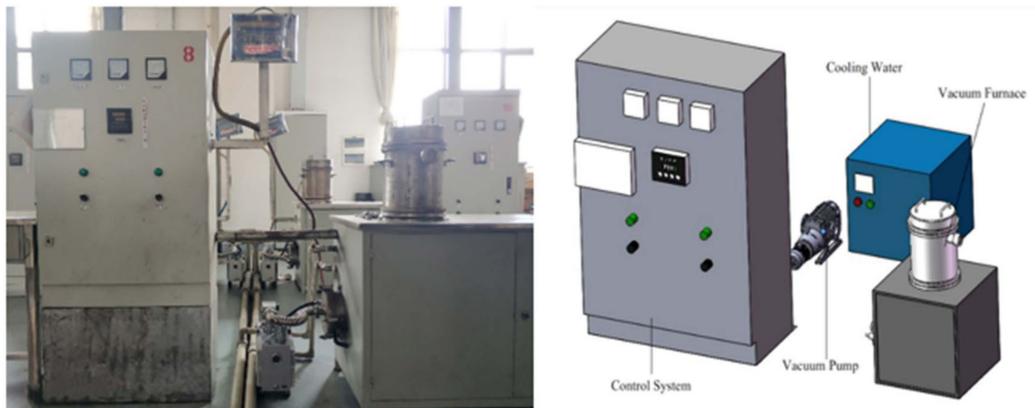
Saturated vapor pressures of oxides increase with escalating temperature at a range of 400 to 1200 K (Figure 3). However, arsenic trioxide's saturation vapor pressure is higher than that of tellurium and tellurium dioxide at the same temperature. For PbO, Sb<sub>2</sub>O<sub>3</sub>, and Bi<sub>2</sub>O<sub>3</sub>, the pressure curves have a shallower slope, which possessed a considerably lower pressure at the same temperature range. Compared with that of TeO<sub>2</sub>, PbO, and Bi<sub>2</sub>O<sub>3</sub> will be non-volatilized and remained in the residues. Therefore, volatilization of tellurium and tellurium dioxide may be accompanied by the volatilization of As<sub>2</sub>O<sub>3</sub> and Sb<sub>2</sub>O<sub>3</sub>.

### 2.3. Experimental Equipment

Experiments were conducted on an internal heating vacuum resistance furnace that was composed of a water-cooling system, an electric heating system, a vacuum system, a control cabinet, etc. (vacuum furnace structure shown in Figure 4). The heating principle involves heating the graphite resistor by radiation generated continuously from the electric current. Radiation heat is transferred to volatile components on the TLAS's surface and then transferred to the inside. The experiment conditions were performed at distillation temperature of 1073–1273 K, constant temperature time of 25–125 min, and system pressure of 10–20 Pa. Tellurium compounds in TLAS will be volatilized into the gas phase, and then be condensed into a solid phase for recovery under a cooling water system's action. The photograph and schematic diagram of vacuum system equipment is shown in Figure 5.



**Figure 4.** A schematic diagram of vacuum furnace structure: 1, water inlet; 2, cooling water; 3, condensate; 4, heating material; 5, graphite crucible; 6, connected to the vacuum pump; 7, raw materials; 8, insulation materials; 9, evaporating utensils; 10, condenser; 11, water outlet.



**Figure 5.** Photograph and schematic diagram of vacuum system equipment.

The specific experimental operations include: (1) raw materials were put into graphite crucible, and the bottom of the crucible was contacted with the thermocouple in a vacuum furnace; (2) vacuum pump was worked to make sure the balance of system pressure 5–15 Pa, and the heating system was turned on, maintained at a specified temperature, and turned off at the ends of gas gasification, respectively; (3) the volatilized products were naturally cooled during the natural water-cooling process, and vacuum pump and cooling water were turned off while the temperature dropped below 473 K; (4) the residuals remained in a graphite crucible and the volatiles condensed in the condenser were collected while the furnace temperature was cooled at a room temperature.

### 3. Results and Discussion

Tellurium was the target separation element of this experimental study. The best parameters for recovering tellurium from TLAS had been obtained through many empirical studies. In addition, the volatilization rate and the direct yield rate of tellurium were calculated by the Equations (7) and (8) to discuss the experimental results.

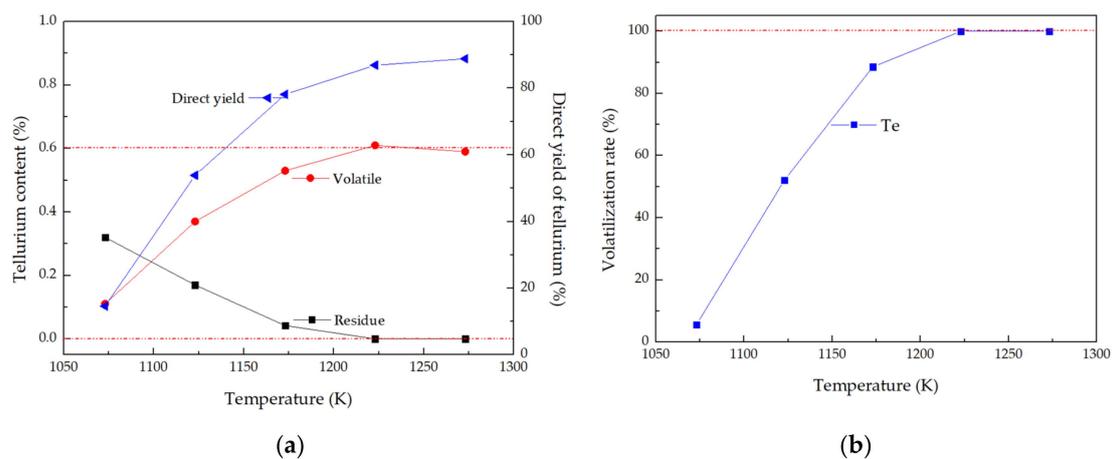
$$\text{Direct yield rate} = \frac{m_1 \times w_1}{m_0 \times w_0} \times 100\% \quad (7)$$

$$\text{Volatilization rate} = \frac{m_0 \times w_0 - m_2 \times w_2}{m_0 \times w_0} \times 100\% \quad (8)$$

where  $m_0$ ,  $m_1$ ,  $m_2$ ,  $w_0$ ,  $w_1$ , and  $w_2$  are the mass of initial material (g), the abundance of volatile (g), the abundance of residue (g), the content of Te in raw material (%), the content of Te in volatile content (%), and the content of Te in residue content (%), respectively.

### 3.1. Temperature Effects on Separation of Tellurium

The effect of distillation temperature (1073 K, 1123 K, 1173 K, 1223 K, 1273 K) on separation of tellurium from TLAS was shown in Figure 6 under the experimental conditions of the system pressure 5–15 Pa and constant temperature time 25 min.



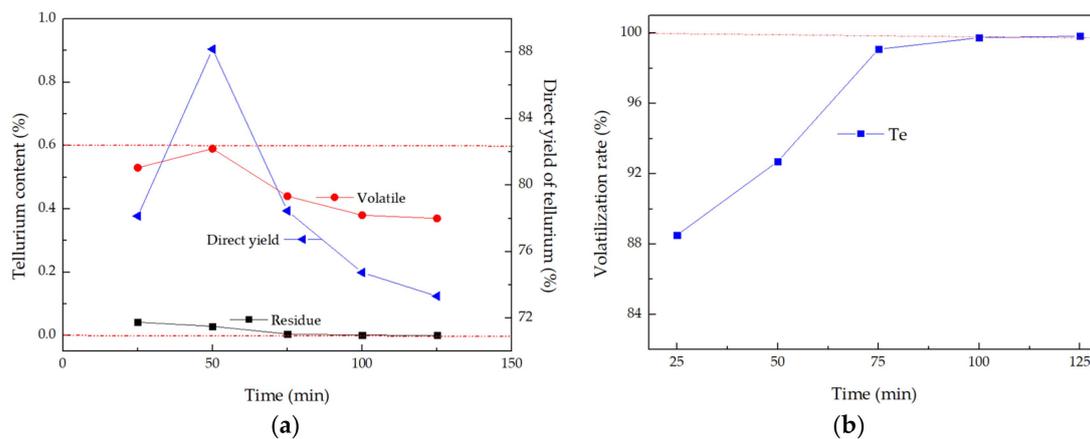
**Figure 6.** Effect of temperature on separation of tellurium from TLAS. (a) content and direct yield, (b) volatilization rate.

The fluctuant curves of the different distillation temperature results (Figure 6) indicate an evident separation of tellurium from TLAS in one step of vacuum distillation under the system pressure of 5–15 Pa and constant time of 25 min. For tellurium in the residues, the curve of the content change has a shallow slope, decreasing from 0.32% to 0.042% at the temperature range of 1073 to 1223 K. The tellurium content in the volatiles gradually increased from 0.11% to 0.62% at the same range. The content of tellurium in the residue was close to 0% at a higher temperature range of 1223 to 1273 K, showing that tellurium had completely volatilized into the gas phase. The tellurium content in the volatiles was maintained at approximately 0.62%. The direct yield of tellurium gradually increased from 15% to 89% at the whole range of 1073 to 1273 K (Figure 6a).

Figure 6b shows the relationship between the volatilization rate of tellurium and distillation temperature. The tellurium's volatilization rate gradually increased with increasing temperature. The tellurium's volatilization rate was close to 100% while the temperature reaches 1173 K, indicating that all tellurium in TLAS have been volatilized into the gas phase and finally condensed in volatile. Therefore, a distillation temperature of 1173 K can be considered as the optimal separation temperature of tellurium from TLAS.

### 3.2. Time Effects on Separation of Tellurium

The effect of constant temperature time (25 min, 50 min, 75 min, 100 min, 125 min) on the separation of tellurium from TLAS was shown in Figure 7 under the experimental conditions of system pressure 5–15 Pa and distillation temperature 1173 K.



**Figure 7.** Effect of constant temperature time on separation of tellurium from TLAS. (a) content and direct yield, (b) volatilization rate.

The fluctuant curves of the distribution rule and direct yield of tellurium (Figure 7a) also indicate an evident separation of tellurium from TLAS in one-step distillation. For tellurium in the volatiles, the curve of the content change has a shallow slope, increasing from 0.52% to 0.61% at the time range of 25 to 50 min. For the direct yield of tellurium, the change was similar, increasing from 78% to 88%. Tellurium in the residues gradually decreased from 0.042% to 0.029% at the same range. However, volatilization and direct yield of tellurium were reversed. The content of tellurium in the volatiles decreased from 0.61% to 0.38%, and direct yield decreased from 88% to 78% at a longer time range of 50 to 125 min. Especially, tellurium content in the residue was close to 0 at 100 min constant.

Figure 7b shows the relationship between the volatilization rate of tellurium and constant temperature time. The tellurium's volatilization rate gradually increased with increasing constant temperature time. The tellurium's volatilization rate was up to 92% when the constant temperature time was higher than 50 min. Tellurium in TLAS has been completely volatilized at a constant time range of 100 to 125 min. Therefore, a constant temperature time of 50 min can be considered as the optimal separation time of tellurium from TLAS based on the balance of direct yield and further energy consumption.

Comprehensive analysis of the influence of distillation temperature and constant temperature time on the separation of tellurium by vacuum distillation of TLAS. The best process conditions for separating tellurium from TLAS: distillation temperature 1173 K, constant temperature time 50 min, and system pressure 5–15 Pa.

### 3.3. Chemical Composition of Tellurium Condensate

Comprehensive composition results of condensate (Table 3) reveal that tellurium has been volatilized along with the gasification of arsenic, antimony, and other substances under the optimal conditions of distillation temperature 1173 K, and constant temperature time 50 min. This gasification phenomenon is consistent with theoretical analysis.

**Table 3.** Chemical compositions of tellurium condensate (wt%).

Component	As	Ag	Te	Bi	Sb	Pb
wt/%	6.9	2.8	3.0	43	35	9.0

It can also be seen (Table 3) that tellurium content in the final condensate is 3%. Tellurium in TLAS was separated and enriched by more than ten times, further indicating that TLAS with high tellurium have been successfully treated by one step of vacuum gasification-directional condensation. However, bismuth and antimony in the final condensate were relatively high, reaching 43% and 35%, respectively. Therefore, the final tellurium condensate can be considered to further extract by means of vacuum gradient

condensation or to synthesize some materials on the strength of multiple requirements of tellurium products.

### 3.4. Environmental Implication for Separation

Vacuum gasification-directional condensation used in volatilization of tellurium was successful in separating tellurium to some degree. It is all known that separation and recovery of tellurium would be beset with difficulties from anode slime.

The traditional treatment process for separating tellurium from lead anode slime consumed various chemical reagents, generated a lot of gas containing toxic substances, and produced waste water and waste residue containing heavy metals, which caused severe damage to the environment and humans, and restricted the ecological development of metal smelting. For example, arsenic-containing gas would cause arsenic poisoning and further migrate to pollute the atmosphere, water, soil, etc. Waste water and waste residues containing heavy metals were difficult to treat and dispose of reasonably.

Nevertheless, the results of vacuum gasification-directional condensation are an important step in separating tellurium and reducing hazardous materials, especially for arsenic-containing water. Vacuum gasification-directional condensation only produced a small amount of soot. Waste residue or waste water containing toxic substances could be completely avoided. This method is environmentally friendly and conducive to the ecological environment's development. In the vacuum treatment process, harmful substances, such as arsenic and heavy metals, will only remain in the residue or condense in the volatile matter, which can be conveniently sourced and directly purified.

## 4. Conclusions

- (1) Vacuum gasification-directional condensation significantly enriched tellurium, and separated both heavy metal copper, and precious metal (gold and silver) into residuals;
- (2) Volatilization rate and direct yield of tellurium gradually increased with increasing temperature, and distillation temperature of 1173 K was the optimal separation temperature of tellurium from TLAS;
- (3) Volatilization and direct yield of tellurium were fluctuant with increasing constant time, and constant temperature time of 50 min was the optimal separation time of tellurium from TLAS;
- (4) Volatilization rate of 92% and direct yield rate of 88% elemental tellurium were achieved under the optimal conditions of distillation temperature 1173 K and constant temperature time 50 min;
- (5) Vacuum gasification-directional condensation was an important step in cleanly separating tellurium and reducing hazardous materials.

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