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Recovery of Tellurium from Waste Anode Slime Containing High Copper and High Tellurium of Copper Refineries

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Abstract: Tellurium is used in cadmium tellurium-based solar cells. Mercury cadmium telluride is used as a sensing material for thermal imaging devices. High-purity tellurium is used in alloys for electronic applications. It is one of the important raw materials for solar energy applications. It is used as an alloying element in the production of low-carbon steel and copper alloys. Tellurium catalysts are used chiefly for the oxidation of organic compounds and as vulcanizing/accelerating agents in the processing of rubber compounds. Even though several researchers tried to recover tellurium from different raw materials, there is no attempt to develop a process flow sheet to recover tellurium from waste anode slime having a high tellurium concentration. In this study, optimum conditions were developed to recover Te and Cu from anode slime with the composition Cu: 31.8%, Te: 24.7%, and As: 0.96%. The unit operations involved are leaching, purification, and electro winning. The optimum conditions for producing Te at a recovery of 90% are found to be roasting of anode slime at 450 °C without the addition of soda ash followed by leaching in 1 M NaOH at 10% pulp density for 2 h. The purity of Te metal achieved was up to 99.99%, which could provide a sustainable energy future. The major impurities of the tellurium are observed to be in the order: Se > Sb > As > Cu.

Keywords: tellurium; leaching; electro-winning; recovery; purity

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1. Introduction

Tellurium is majorly used as an alloying element [1] in the production of free-machining low-carbon steel and copper alloys to enhance its machinability without altering the conductivity. Tellurium-based catalysts are mainly employed for hydrogenation, oxidation, and halogenation reactions of organic compounds. Tellurium compounds are applied in the processing of rubber-based compounds as vulcanizing, accelerating agents, and batteries [2]. It has also been widely used in cadmium tellurium-based solar cells [3]. Tellurium, as mercury cadmium telluride, is utilized in thermal imaging applications as a sensor material. Tellurium in its very pure form is used for electronic applications. Large-scale commercial production of CdTe solar panels and Bi_2Te_3 in refrigeration technologies in recent years has significantly increased the demand for tellurium [4]. The world reserves of tellurium are 24,000 tons contained mostly in copper resources. The average world production of tellurium is estimated at 450–500 tons per year [5]. Tellurium is majorly produced in the United States, Peru, Japan, and Canada. Processing 500 tons of copper ore usually produces 1 pound (0.45 kg) of tellurium. Tellurium's main source is the anode slime produced during the electrolytic refining of blister copper. Tellurium and selenium were separated using the copper cementation technique [6].

Halli et al. [7] studied the electrochemical recovery of tellurium from metallurgical industrial waste. The highest tellurium recovery through pregnant leach solution via

electrowinning is 55 wt% purity in the produced deposits, whereas electro-deposition followed by spontaneous redox replacement is 64 wt%. The processing of cemented telluride has been carried out through a hydrometallurgical process with minimal discharge from copper refining. Rheea et al. [8] recovered tellurium through a series of unit operations involving leaching, precipitation, and electro-winning steps. Yue et al. [9] fabricated a hierarchical hybrid membrane for efficient recovery of tellurium from photovoltaic waste.

Li et al. [10] studied the leaching behavior of metals via an alkali fusion-leaching route from copper anode slime for the preliminary separation of metal values. Under optimal conditions, leaching recoveries of nearly 97% Se, 98% As, 86% Sn, and 76% Pb were achieved under optimal conditions leaving the platinum, silver, gold, and tellurium values in the residue.

Gómez-Gómez et al. [11] synthesized tellurium-based nanoparticles and concluded that Te nanoparticles strongly affect biofilm biomass. An efficient and cost-effective radiochemical technique was developed by Sankha Chattopadhyay and Sujata Saha Das [12] to separate tellurium from reactor-irradiated TeO_2 targets with good yield and high quality.

Jin et al. [13] studied the electrochemical recovery of fine tellurium powder from hydrochloric acid media via mass transfer enhancement. A turbulent cylindrical reactor having a large surface area cathode was employed for the recovery of tellurium as microscale Te powder from dilute solution with a recovery rate of 96.1% and a current efficiency of 84.3% while effectively suppressing the undesired reactions of chlorine generation and TeO_2 production. Fan et al. [14] found out the conditions for sodium hydroxide leaching of tellurium with a solid to liquid ratio of 1:6 and free caustic concentration in a solution of 30–40 g/L, with a temperature of 120 °C. Fan et al. [15] extracted high-purity bismuth along with tellurium while treating the residue of zinc anode slime by sulfation roasting, leaching, and electro-deposition processes.

Guo et al. [16] have investigated the recovery of Te from high tellurium-bearing resources by leaching using alkaline sulfide followed by sodium sulfate precipitation. More than 90% Sb and Te leaching efficiency was achieved under optimal conditions. Furthermore, with respect to other metals, such as antimony, arsenic, and cadmium, leaching and releasing studies have been reported by several authors [17–20], which is summarized briefly in this section. Antimony release characteristics of blast furnace slag, mining waste rock, and tailing sand were investigated in static immersion and dynamic leaching tests by Ren et al. [17] and they concluded that the Sb release capacity of the three samples was in descending order of tailing sand, blast furnace slag, and mining waste rock. Zhang et al. [18] studied the leaching and releasing characteristics and regularities of antimony and arsenic from antimony mining waste rocks, blast furnace slags, and tailings. It was suggested that a lower solid–liquid ratio and small particle size improve the leaching efficiency. The concentration of heavy metals, chemical speciation, and leaching characteristics of antimony ore tailings was analyzed by Zhou et al. [19]. The results show that the concentrations of Sb, As, and Hg in the leachates increased with increasing solid–liquid ratio, decreasing particle size, and increasing temperature. Meng et al. [20] aimed to identify the microbes involved in regulating Cd solubility and reveal possible mechanisms. It has been reported that anaerobic microbes, such as anaerolineaceae played an important role in shaping the microbial community in soil and regulating the Cd solubility.

The continuous removal and recovery of tellurium in an up-flow anaerobic granular sludge bed reactor were reported to recover Te from very dilute solutions (Mal et al. [21]). Recovery of tellurium from de-copperizing leach solution of copper refinery slimes by a fixed bed reactor packed with copper cuttings was studied [22]. This study showed to enhance the recovery of copper telluride from a de-copperizing leach solution at 80–90 °C temperature and linear velocity of 3.7 cm/s. Recycling of copper telluride from copper anode slime processing was reported by Xu et al. [23] and Xu et al. [24]. Tellurium and copper were selectively recovered from copper telluride through hydrothermal leaching under the optimized conditions of 5 mol/L NaOH, 5:1 liquid to solid ratio, 150 °C temperature, and 2 h reaction time. During this process, copper was enriched in the solid residue as copper

oxide, and more than 95% of tellurium was selectively dissolved in the solution. Recovery of tellurium from aqueous solutions by adsorption with magnetic nanoscale zero-valent iron is reported by Yu et al. [25] and from chloride solutions using tri-iso-octylamine by Mandal et al. [26].

Even though several researchers tried to recover tellurium from different raw materials to the best of our knowledge, there is no attempt to develop a process flowsheet to recover tellurium from waste anode slime having high tellurium concentration. In this study, optimum conditions were developed to recover Te and Cu from anode slime having the following composition Cu: 31.8%, Te: 24.7%, and As: 0.96%, towards a sustainable copper mining industry.

2. Experimental

2.1. Materials

The anode slime processed in the present work has been collected from the copper refineries of India. All the chemicals, such as NaOH, Na_2CO_3 and H_2O_2 of 99.9% purity, were purchased from Merck, India.

2.2. Method

2.2.1. Leaching

The leaching of anode slime was carried out using a 1 L capacity 316 SS autoclave with two six-blade turbine impellers. The autoclave has a provision for a gas inlet/outlet, internal cooling coil, PID controller for precision temperature control, and digital display of temperature and agitation. At regular intervals, samples were collected by opening the sampling valve connected to a dip tube inserted inside the leaching solution. Leaching recovery is calculated from the weight ratio of Te in the leach liquor to Te content in the anode slime.

2.2.2. Roasting of Anode Slime

The roasting was carried out in a muffle furnace. Sodium carbonate was mixed properly with the powdered anode slime in a quartz crucible. The crucible was then kept in the furnace. Subsequently, the furnace was heated (between 350 to 550 °C) and roasting was carried out for a fixed duration followed by controlled cooling.

2.2.3. Electrowinning of Te

The electrolytic cell used in these experiments was a 250 mL plastic beaker. One cathode (stainless steel 304) and one anode (stainless steel 304) were placed 2.5 cm apart in the cell. DC voltage was applied from a rectifier for initiating the electrolysis operation. After the electrolysis, the cathode and the anode were removed, and the electrolyte was filtered. The Te powder was then washed with distilled water till the filtrate water is completely free from alkali which was tested by measuring its pH using a pH meter. Then, the Te powder was washed with acetone for fine particles and dried. The current efficiency was calculated from the ratio of the actual weight of Te deposited during electrolysis to the theoretical weight of Te to be deposited with respect to the applied current for the experimental period of time.

3. Results and Discussion

3.1. Alkali Leaching of Anode Slimes

The dissolution of copper telluride in alkaline solution may be represented by the following Equation (1):

$$Cu_2Te + 2NaOH + 2O_2 = 2CuO + Na_2TeO_3 + H_2O \quad (1)$$

From Equation (1) it is observed that 1 mole of tellurium will require 2 moles of alkali. Considering the tellurium content it can be said that the maximum tellurium for 10%

pulp density slurry will be 0.12 M, hence 0.24 M will be the stoichiometric requirement. Considering the above points, further experiments were carried out using 0.5 M NaOH for 5% slurry or 1 M NaOH for 10% slurry at ambient temperature.

After analyzing the results with the sample, it was decided to concentrate on the alkali leaching step to recover tellurium value because once tellurium is extracted copper leaching will be an easier task.

3.1.1. Preliminary Leaching Test

A preliminary leaching test of the sample having high copper and high tellurium was carried out under the condition of 10% pulp density (termed PD) and 1 M NaOH. The results are given in Table 1.

Table 1. Results of preliminary leaching study, time 2 h.

S. N.	Leaching Conditions			Te Recovery (%)
	Temperature	Pulp Density (%)	NaOH Conc., M	
1	Ambient	10	1	36.8
2	80 °C	10	1	64.7

It is evident from Table 1 that under the same conditions of pulp density and alkali conditions, leaching responses are comparatively less. Even at elevated temperatures, only 64% tellurium recovery was obtained.

3.1.2. Alkali Leaching under Oxidizing Conditions

To improve the tellurium recovery, an attempt was made to impose oxidizing conditions during leaching using both hydrogen peroxide as well as oxygen under pressure. The results obtained under different conditions are presented in Table 2.

Table 2. Tellurium recovery under oxidizing conditions, Time 2 h.

S. N.	Leaching Conditions		Te Recovery (%)
	Temperature	Oxidant	
1	Ambient	H_2O_2: 5% (*v*/*v*)	50.3
2	Ambient	H_2O_2: 10% (*v*/*v*)	56
3	80 °C	O_2: 2 kg/cm^2	68.2
4	90 °C	O_2: 2 kg/cm^2	73.1
5	125 °C	O_2: 2 kg/cm^2	73.7

Increasing the temperature in oxidizing pressure leaching has not improved the recovery beyond 73% which establishes the complex nature of the material.

3.1.3. Soda Ash Roasting-Alkali Leaching

Roasting of the anode slime was carried out using sodium carbonate (Na_2CO_3) as a roasting agent. After carrying out roasting at temperatures from 350 to 650 °C for 2 h in the presence of 0.2 g Na_2CO_3/g of the anode slime sample, the roasted mass was cooled and leaching was carried out in 1 M NaOH solution. The results of soda ash roasting–alkali leaching obtained are presented in Figure 1.

It is observed from Figure 1 that with an increase in roasting temperature, there is an increase in tellurium recovery up to 450 °C after which there is a significant decrease in recovery. This may be due to the phase formation of sodium tellurate (Na_2TeO_4) which is not water-soluble whereas another form of sodium tellurite (Na_2TeO_3) is highly soluble.

3.1.4. Roasting without Soda Ash

After observing the highest recovery obtained when soda roasting was carried out at 450 °C. The next set of roasting at the same temperature was carried out without adding

any Na_2CO_3. Roasted material is leached and the recovery efficiency is 90%. Although 98% Te recovery was possible by roasting in the presence of 20 wt.% Na_2CO_3 it is possible to get about 90% Te recovery without using any Na_2CO_3.

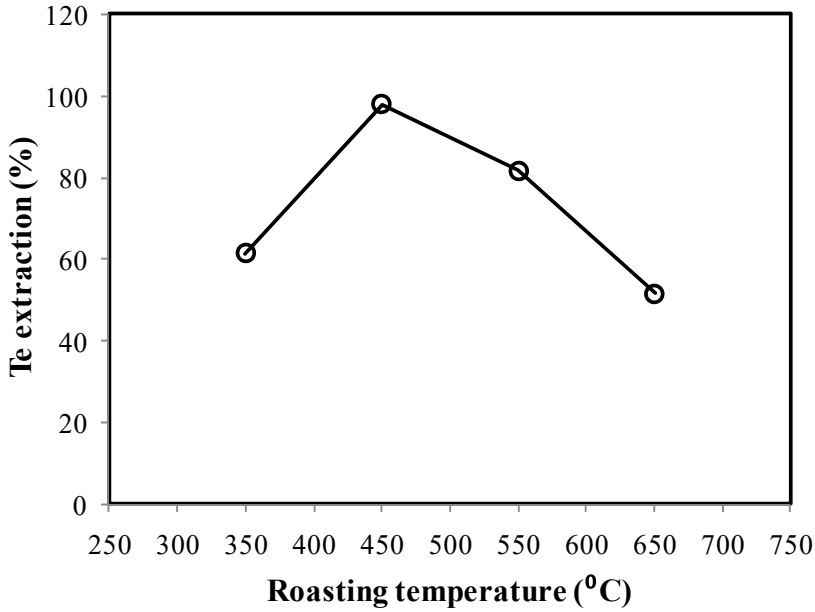

Figure 1. Effect of roasting temperature on tellurium recovery (Conditions: Time 2 h, Na_2CO_3 0.2 g/g sample. Leaching conditions are at ambient temperature, 10% P.D., 1 M NaOH).

3.1.5. Leaching in Recycled Spent Electrolyte

To recycle the spent electrolyte to be generated from Te electrolytic cells leaching test was carried out in spent electrolyte for four cycles. About 200 g of the sample was roasted at 450 °C to generate the roasted mass for all the leaching experiments carried out later.

Then, leaching was carried out under the standard conditions of 1 M NaOH and 10% pulp density at ambient temperature for 2 h. This leach liquor was subjected to electrowinning for Te powder production and the spent electrolyte (S.E.) generated from this leach liquor was used as a leaching medium in the next stage after adding the required make-up amount of NaOH to adjust the concentration of solution to 1 M NaOH. The results are shown in Table 3.

Table 3. Leaching conditions 1 M NaOH, 10% P.D., 2 h.

Cycle No.	Te in S.E, g/L	Free Alkali in S.E., g/L	Concentration (g/L)		Te Pick Up from Solid, (g/L)	Te Recovery, (%)
			Te	Se		
0	-	40	24.9	2.16	-	-
1	11.2	24	28.8	1.9	17.6	63.81
2	7.13	24	29.4	1.32	22.27	80.75
3	5.7	22	29.1	1.12	23.4	84.84
4	7.1	24	28.2	1.8	21.1	76.5

The spent electrolyte can be recycled effectively; however, a decrease in Te recovery was observed which may be attributed to the build-up of other ions. Moreover, a higher initial concentration of Te in spent electrolyte is also observed to affect the leaching efficiency adversely. Therefore, the electrowinning operation may be carried out to an extent of Te concentration depletion such that the spent electrolyte contains up to 5 g/L Te or less.

In the present work, considering the complex nature of the anode slimes, different leaching conditions, such as alkali leaching, alkali leaching under oxidizing conditions, soda ash roasting–alkali leaching, and roasting without soda ash-alkali leaching have been

experimented. Because of varied leaching conditions, the leaching recovery of Te has changed significantly. Moreover, in the cyclic tests using a spent electrolyte, the fluctuation in Te recovery may be due to the variation in Te concentrations of spent electrolyte and roasted mass feed.

3.2. Electrowinning of Tellurium

The liquor obtained from solvent extraction contained some organic solvent. Therefore, this liquor was treated for adsorption for about 1 h using activated charcoal.

3.2.1. Effect of Current Density

The purified electrolyte was then tested for producing Te powder. The results of such tests are presented in Table 4. Current density varies from 50 A/m^2 to 250 A/m^2 and it is found with an increase in current density, current efficiency is reduced (Figure 2) and energy consumption is increased (Table 4). It is observed from the results presented in Table 4 that the current efficiency decreased with increasing the current densities. The deposition was greater at the current density of 150 A/m^2.

Table 4. Variation of current density.

Current Density (A/m^2)	Voltage (V)	Current Efficiency (%)	Energy Consumption (kWh/kg)
50	1.7	85.4	1.67
100	2.1	85.17	2.07
150	2.2	84.3	2.19
200	2.5	70	2.93
250	2.25	62.23	3.03

Conditions for Electrowinning: Conc. of Te—15 g/L, NaOH—21.4 g/L, Temperature—ambient temperature (32 ± 3 °C), Duration—6 h.

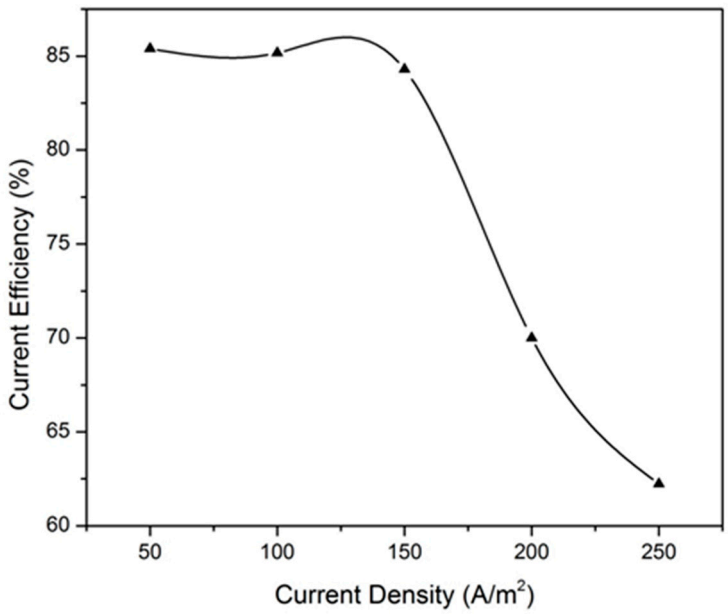

Figure 2. Effect of current density on current efficiency (Conditions for Electrowinning: Conc. of Te—15 g/L, NaOH—21.4 g/L, and Duration—6 h).

Purity of Te Powder

The digestion of Te metal was first analyzed by atomic absorption spectroscopy to detect the contamination of Te with probable metallic impurities. From the results, it is observed that the major impurities are Se, As, Cu, and Sb. Then, the chemical analysis for these impurities is carried out, the results of which are given in Table 5. The purity of Te

metal achieved was up to 99.99%. The major impurities of the tellurium are in the order: Se > Sb > As > Cu.

Table 5. Analysis of Te powder.

Current Density (A/m^2)	Impurities (%)				Total Impurities (%)	Purity of Te Metal (%)
	Cu	As	Sb	Se		
50	0.0042	0.0	0.0081	0.055	0.067	99.93
100	0.003	0.0	0.066	0.065	0.134	99.86
150	0.0058	0.0	0.0007	0.0071	0.0137	99.99
200	0.0032	0.0332	0.004	0.0138	0.0542	99.95
250	0.0039	0.0196	0.02	0.255	0.2985	99.7

Conditions for Electrowinning: Conc. of Te—15 g/L, NaOH—21.4 g/L, Temperature—ambient temperature (32 ± 3 °C), Duration—6 h.

3.2.2. Effect of Tellurium Concentration in Electrolyte

The electrolyte was first purified from carried-over solvent by treating it with activated carbon (charcoal powder). The electrolyte concentration varies from 15 g/L to 25 g/L and it is found with an increase in concentration, current efficiency is increasing (Figure 3). It is observed from the results presented in Table 6 that the Te metal can be electrowon with more than 87% current efficiency.

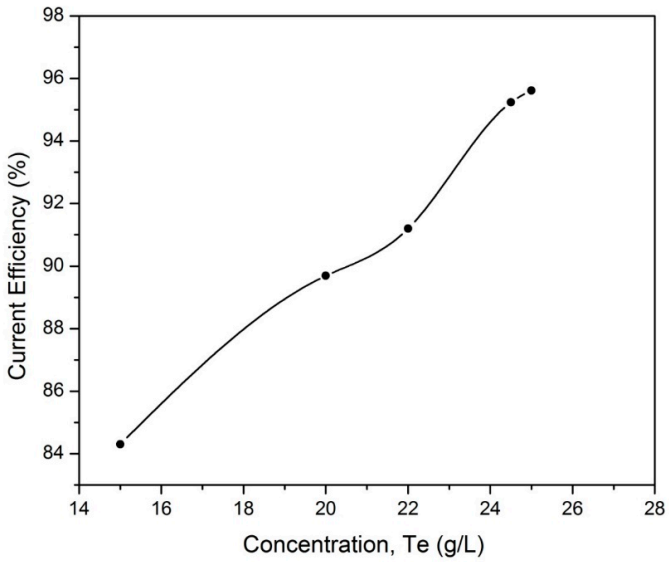

Figure 3. Effect of concentration of tellurium on current efficiency (Conditions: Current density 150 A/m^2, NaOH—21.4 g/L, Duration—6 h).

Table 6. Variation of conc. of tellurium in the electrolyte.

Conc. of Te in Electrolyte, (g/L)	C.V, (V)	E.C (kWh/kg)
15	2.20	2.10
20	2.10	2.00
22	2.10	1.95
24.5	2.00	1.76
25	2.00	1.72

Conditions: Current density—150 A/m^2, Duration—6 h, Concentration of NaOH—21 g/L.

Purity of Te Powder

The Te powder was analyzed by atomic absorption spectroscopy to detect the contamination of Te with metallic impurities. The results are given in Table 7. The purity of Te achieved was 99.9%. The contamination of the cathode due to various impurities are in the order of Se > As > Sb > Cu.

Table 7. Analysis of Te powder.

Te Conc. (g/L)	Impurities (%)				Total Impurities (%)	Purity of Te Metal (%)
	Cu	As	Sb	Se		
24.9	0.0002	0.02	0.003	0.076	0.09	99.9
28.81	0.0007	0.022	0.007	0.25	0.27	99.72
29.4	0.0005	0.022	0.005	0.39	0.42	99.57
29.14	0.0015	0.02	0.004	0.45	0.48	99.52
26.21	0.0014	0.02	0.005	0.66	0.68	99.31

3.3. Acid Leaching of Residue

In the second stage, sulphuric acid leaching was carried out for the dissolution of copper from the alkali leach residue. Sulphuric acid leaching was carried out at ambient temperature for the two residues collected from 50 g and 100 g alkali leaching experiments. The results on Cu recovery from the two experiments are given in Table 8.

Table 8. Extraction of copper from alkali leach residue.

S. N.	Leaching Conditions			Cu Recovery (%)
	Residue wt. (g)	Pulp Density (%)	H_2SO_4 Conc., M	
1	35.5	10	1.1	86
2	70	10	0.94	90

Higher recovery of copper from the residue as compared to original anode slimes is due to the conversion of copper telluride to copper oxide which is more easily acid soluble. The copper from the leach liquor is processed through electrowinning. The process flow sheet for the recovery of tellurium and copper is given in Figure 4. This study summarizes that the copper supply chain can supply tellurium needs for the near term, for which a significant amount must be captured during processing. This process seems to be more economical as the costly unit operation of solvent extraction is avoided in developing the process flowsheet of the technology.

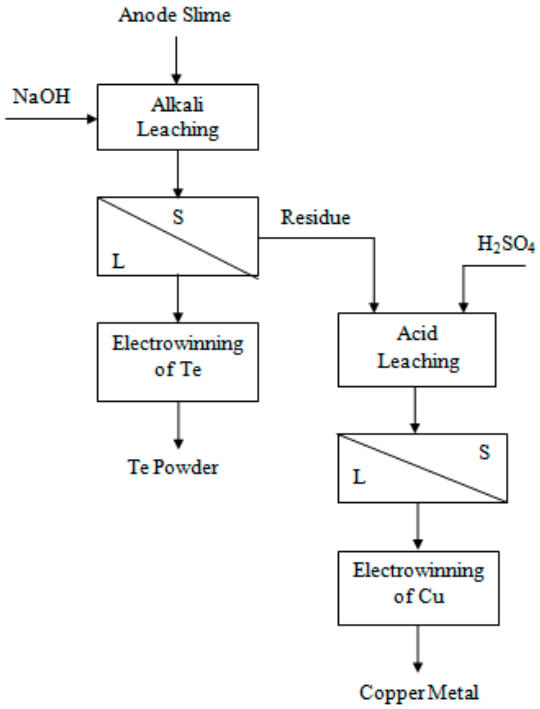

Figure 4. Process flowsheet for production of Te and Cu from anode slime.

4. Conclusions

The following conclusions are summarized from the present study.

- The room temperature leaching, as mentioned in method 1, resulted in lower recovery (~37%). High-temperature leaching in the presence of oxygen pressure in 1 M NaOH could give a recovery of ~70%.
- Roasting of anode slime for 2 h at 450 °C with 20% $NaCO_3$ addition followed by ambient temperature leaching in 1 M NaOH could result in more than 95% Te extraction.
- The most efficient conditions for extraction of Te are air roasting at 450 °C without any addition of soda ash followed by leaching under the conditions: 1 M NaOH, 10% pulp density, 2 h. Te recovery was found to be ~90%.
- The use of spent electrolytes in the leaching of roasted samples has established the possibility of utilization of spent electrolytes in a preferable way if the spent electrolyte contains 5 g/L or less tellurium.
- As the demand for Te is increasing day by day, it will be better to treat anode slimes for the production of Te from secondary resources. Therefore, the findings of the research should lead to commercialization of the process.

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