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Full Characterization of Sarcheshmeh and Khatoon-Abad Copper Anode Slimes: Characterization Impact on the Decopperization Operation

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A B S T R A C T

Copper anode slime, a valuable by-product generated during the electrorefining process, is an important secondary resource for the recovery of metals, such as gold, silver, selenium, tellurium, PGMs and copper. A full characterization of the anode slime is an essential part of the efficient recovery of these valuable metals. In this research, the refinery slimes of the Sarcheshmeh and Khatoon-Abad copper complexes, were fully characterized and compared using inductively coupled plasma (ICP), X-ray diffraction (XRD), X-ray fluorescence (XRF), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), thermogravimetric analysis (TGA), differential thermal analysis (DTA), and particle size analysis. Besides the bottom cell slime characterization, properties of the Sarcheshmeh's adherent slime was also analyzed and correlated. Sarcheshmeh copper slime particle size was found much smaller than the Khatoon-Abad slime. For both slimes though, the major part the gold and tellurium are located in larger than 38-micron particles. The main difference in thermal analysis of the two slime samples was originated from their selenium content difference. This has caused the higher concentration of non-selenide copper compounds at Khatoon-Abad slime, resulted in its easier copper recovery. Decopperization of both bottom-cell slimes were investigated and a significant difference in copper dissolution behaviors of the two slimes were observed due to their characteristic difference including the copper-selenide phases and capsulated copper in the barium sulfate agglomerates. Copper dissolution from Khatoon-Abad slime was observed to reach 90% comparing to 40% for Sarcheshmeh slime using atmospheric leaching.

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1. INTRODUCTION

In the copper electrorefining process, the impure anode electrochemically dissolves and deposit as a pure metal on the cathode surface. During this process, a solid by-product of the anode slime is generated. The slime generally contains Cu, Ag, Au, Se, Te, Pb, Ni, Sb, As, and elements from platinum group metals [1, 2]. The composition of the slime depends on the input concentrate and the extraction method [3, 4]. Therefore, the slime analysis and its properties vary from one refinery plant to another. Moreover, the mineralogy and the composition of the slime may also drastically change within the same plant due to the fluctuation of the feed material and the process disturbances [4]. Copper anode slime is a dark-colored, fine-grained powder with the particle size often less than 50 microns with the spherical, semicircular, and elongated plate shapes [5-12]. Slime production usually varies from 2 to 28 kg per ton of copper cathode with an average value of 5 kg/ton at Sarcheshmeh and Khatoon-Abad [13]. The precious metal content of the anode is concentrated over the electrorefining process and collected in the slime. The small particle size of the slime makes some of the operating cost contributors, such as crushing and grinding a virtue out of necessity. Comprehensive studies were done by Chen and Dutrizac [5-12, 14, 15] on the characterization of copper anode slime, which shows that precious metals are mainly present in the oxide complexes and selenide phases. Anode slime constituents

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are often transferred from the anode to the slime in various ways. During the copper electrorefining process, elements with less reduction potentials than the copper dissolve from the copper anode, whereas elements with higher reduction potential than copper are deposited at the bottom of the electrolyte cell [16].

Anode slime is classified into two main groups: the primitive slime, which is mainly collected at the bottom of the electrorefining cells, and the secondary slime, which mainly forms on the anode surface or suspended in the electrolyte [17]. The slime is continuously formed during the electrorefining process. Wang et al. [18] represented the classification of anode slimes, according to Figure 1.

Primitive slime contains metals nobler than copper, such as gold, and other insoluble compounds, such as selenides, telluride, or other complex refractory oxides. The components of this slime are directly released from the slime and mostly fall to the bottom of the electrolyte cell. A portion of this slime can also coat the surface of the copper anode as a porous layer. Secondary slime includes compounds precipitated in the interface of the anolyte region (generates by the 1st reaction) where other phases formed from the electrolyte (2nd reaction). For example, lead sulfate is a 1st reaction slime component formed by the reaction of Pb^{2+} (dissolved from the copper anode) and sulfate. Elements such as Ni, As, Sb, Bi, and Ag are partially soluble in the electrolyte; therefore, they can be found in both slime and electrolyte. Compounds of these elements mainly belong to the 2nd reaction slimes. Insoluble compounds of these elements, such as Kuferglimmer, can report to the primitive slime. However, gel-like phases containing these elements are named floating slime and are usually suspend in electrolyte. In copper refinery plants, barium sulfate is added to prevent the adhesion of the cast anodes to the mold. Excessive use of $BaSO_4$ could result in contamination of the anode and, consequently, the primitive slime. The presence of barium in the slime can adversely affect the extraction efficiency of the precious metal.

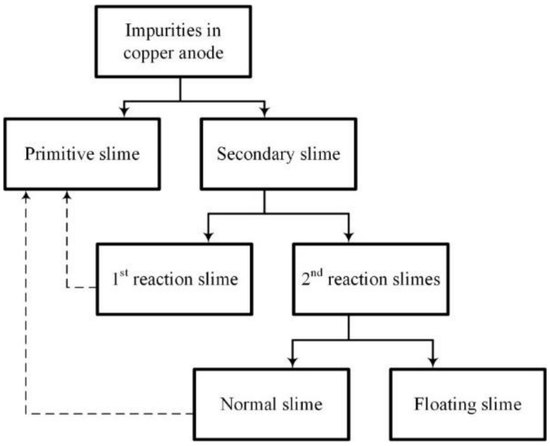

Figure 1. Classification of different types of copper anode slime [18]

Sarcheshmeh copper complex and Khatoon-Abad copper complex as the two largest copper producers of the country are producing about a total of 250,000 tons/year copper cathode [19]. A considerable number of studies have been conducted on the recovery of the metallic content of the Iranian copper anode slimes, mostly from Sarcheshmeh copper complex due to its earlier operation history. The authors of this work have found about 40 research works on the recovery of the Sarcheshmeh copper slime mostly written in Farsi. None of the studies were suggested a comprehensive plan for the recovery of all recoverable constituents of the slime, including copper, selenium, tellurium, silver, gold, and PGMs. Few studies were focused on the recovery of the two or, at most, three of the elements. For instance, Dehghanpoor et al. [20] investigated the extraction of Au and Cu from Sarcheshmeh bottom-cell slime. The result showed that by using a multi-stage leaching process in sulfuric acid, nitric acid, and aqua regia, more than 90% of the Cu and 80% of the Au could be dissolved from the slime. Similarly, Khanlarian et al. [21] used a sulfation-roasting process for recovering Cu, Ag, and Se content of the Sarcheshmeh bottom-cell slime. At their optimum experimental conditions, (sulfation at T=250°C, and acid to solid ratio of 1.95 followed by leaching in water), more than 90% of the stated metals were observed to be recovered. Saeedi et al. [22] studied the dissolution behavior of the Au from Sarcheshmeh bottom-cell slime by acidic chloride leaching at elevated temperature. Under the optimum conditions, the recovery of more than 90% of gold was shown to be obtained. Few more examples of the published researches on the recovery of the metallic content of the copper anode slime mostly from Sarcheshmeh Copper Complex are listed in literature [23-29]. Despite the extensive research have conducted on the recovery of the metallic content of the refinery slimes, none has made a comprehensive study in characterization of different types of slimes. Moreover, the main part of the available work on characterization of the copper anode slimes, date back to at least twenty years ago. Accordingly, recent variations in copper ores and the changes applied in the copper production processes, that can potentially alter the anode slime characterization, has been another impetus to conduct this study. In this research, various analysis techniques have been used to characterize and compare various types of the Sarcheshmeh and Khatoon-Abad anode slimes

The aim of this research is to provide a vision on the recovery plan of the various types of slime based on their characteristics. To recover the valuable content of the slime and to propose a comprehensive recovery flow diagram, the elemental analysis, morphology and microstructure, phase analysis, particle size, and thermal analysis of the slime should be determined. These characteristics can strongly influence the selection of the proper method of recovery for valuable content of the slime. For better illustration of the importance of the characterization, decopperization of the bottom-cell slimes of Sarcheshmeh and Khatoon-Abad, considering the characterization results were investigated and described. Several procedures were suggested in removal of copper from the copper anode slime, including atmospheric sulfuric acid leaching, sulfuric acid roasting, atmospheric oxidation acid leaching with air and oxygen and pressure oxidative sulfuric acid leaching [30, 31].

The pressure oxidative sulfuric acid leaching is a simple and fast process that generates very high copper recovery of copper, but high reactor corrosion alongside low leaching efficiency of selenium and tellurium [32, 33]. In atmospheric oxidative sulfuric acid leaching, copper is oxidized to copper sulfate in a dilute sulfuric acid solution using O_2 gas. This is an easy, cheap and simple process with low consumption of H_2SO_4. The main drawback is the partial oxidation of cuprous sulfide and selenide that causes typical recovery of copper blow 80%. The aim of this research, apart from the full characterization, was to deliver a vision on differences of various types of slime and its impact on the recovery of the constituent metals.

2. EXPERIMENTAL PROCEDURE

Two different types of anode slime were characterized in this research. The first type of slime was gathered from the anode surface, which is known as the adherent slime. The second type of slime was gathered from the bottom of the electrorefining cells, which is known as bottom-cell slime. Both Sarcheshmeh bottom-cell and adherent slimes were characterized; however, in the case of Khatoon-Abad, only bottom-cell slime was characterized. The chemical compositions of the slimes were determined using the fire assay analysis (for gold and silver), inductively coupled plasma optical emission spectroscopy (ICP-OES, Varian735), inductively coupled plasma mass spectrometry (ICP-MS, HP4500), and X-ray fluorescence (XRF, ARL8410). The particle size distribution of the bottom-cell slime was determined by screen analysis. X-ray diffraction (XRD, Philips PW 3040/60) was used for phase detection. The microstructure of the samples was studied using an electron microscopy (SEM, FEI Quanta 450) equipped with an energy dispersive spectroscopy (EDS, Brunker Xflash 6L10). Simultaneous thermogravimetric analysis (STA, PerkinElmer STA6000) with a heating rate of 10°C/min from 30 to 800°C, was used for thermal characterization of the bottom-cell slimes. The chemical composition of the Sarcheshmeh electrolyte was also determined using ICP-OES technique. A scheme of the methodology used in this study is shown in Figure 2. Finally, decopperization of Sarcheshmeh and Khatoon-Abad bottom-cell slimes were studied based on the obtained results of the characterization section. Leaching experiments for the recovery of copper were conducted at T=85°C, sulfuric acid concentration of 160 g/L, solid to liquid ratio of 1:5 g:ml and in presence of air and pure oxygen.

3. RESULTS AND DISCUSSION

3. 1. Chemical Composition Results

The chemical compositions of Sarcheshmeh bottom-cell slime (SBS), Sarcheshmeh adherent slime (SAS), Sarcheshmeh electrolyte, and Khatoon-Abad bottom-cell slime (KBS) are shown in Table 1. As evident, notable differences can be observed between various types of slime within a refinery plant (Sarcheshmeh). Also, there are distinct differences between the slimes of different copper refinery plants.

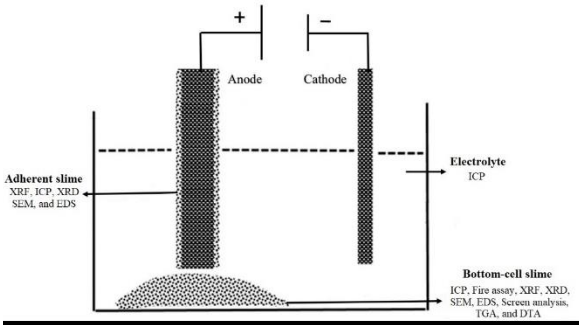

Figure 2. Scheme of the methodology used for characterizing the samples

TABLE 1. Chemical composition of SBS, SAS, Sarcheshmeh electrolyte and KBS

Elements	SBS (wt.%)	SAS (wt.%)	Sarcheshmeh Electrolyte (ppm)	KBS (wt.%)
Cu	8.18	34.74	40110	23.47
Ag*	6.5	7.78	0.005	2.11
As	1.03	4.37	3068	3.10
Au	0.15	-	-	0.11
Ba*	20.90	0.41	0.09	6.71
Ni	0.02	0.01	812	0.04
Pb	9.02	11.93	5.4	2.00
Sb	0.70	0.72	236	0.87
Se	14.09	16.29	0.7	7.73
Te	0.15	0.85	0.01	0.59

* Measured by XRF

Regarding Sarcheshmeh slime, copper concentration is considerably higher in the adherent slime in comparison to the bottom-cell slime. The adherent slime forms on the surface of the copper anode, which is mainly constituent of the metallic copper, copper oxide, and copper sulfate. Moreover, the main portion of the copper constituent of the bottom-cell slime is dissolved, whether during the electrorefining process by the electrolyte turbulence or through the collection of the slime process, which could result in a lower copper concentration. The Sarcheshmeh bottom slime was collected after the slime was water washed at the electrolysis cell and passed over through the collection tanks, thickeners, and the filter press. It should be noted that, most of the copper finds its way to the bottom-cell slime. Accordingly, despite the higher copper concentration of the adherent slime, a higher fraction of the total copper is presented in the bottom-cell slime. The Khatoon-Abad slime is, however, collected right after the electrolysis cell. The Khatoon-Abad slime wash with 80°C deionized water at S/L ratio of 1/4 g/mL over 2 hours was proven the weight loss of about 30%. The prior wash and after wash analysis of the Khatoon-Abad slime is shown in Table 2. The solubility of a few constituents of slime such as arsenates and entrapped copper sulfate are much easier in hot water than the sulfuric acid, copper sulfate solution medium.

Another significant difference between the adherent slime and the bottom slime is the barium concentration. Most of the barium is dropped down to the bottom cell due to its larger particle size and higher density. The barium concentration in the Sarcheshmeh adherent slime is negligible compared to its concentration in the Sarcheshmeh bottom-cell slime. Some elements, such as As, Sb, Se, and Ni, have been found in both of the slimes, as well as, in the electrolyte. These are the partially soluble elements that are expected to be found in the slimes and the electrolyte. The concentrations of the partial soluble elements are a little higher in the Sarcheshmeh adherent slime. This is possibly due to the dissolution of a fraction of these elements during their transformation to the Sarcheshmeh bottom-cell slime. Pb is found in both Sarcheshmeh bottom-cell slime and Sarcheshmeh adherent slime in an almost similar concentration. For the lead, non-soluble or low-soluble species of the element is formed in the sulfuric acid medium. This is, however, after the lead is being dissolved in a cationic form from the anode. The dissolved lead is rapidly reacting with the sulfate content of the solution and forms the lead sulfate layer on top of the anode. This layer is partly dropped to the cell bottom but continuously generates on the anode surface. The Pb behavior is different from the barium as the barium sulfate particles are non-soluble and originate from the barium sulfate slurry added through the casting process. The barium sulfate particles are adherent to the anode surface, and by the dissolution of the copper content of the anode is dropped form the anode surface to the cell bottom. The adherent slime was found free of the barium sulfate as the barium may just appear at the beginning of the dissolution process due to its presence on top of the anode surface .

TABLE 2. Chemical composition of Khatoon-Abad bottom-cell slime before and after washing with 80ºC water

Element	Concentration before washing (%)	Concentration after washing (%)	Recovery (%)
Cu	21.47	24.56	19.94
Ag	2.11	3.01	-*
As	3.10	3.42	22.84
Au	0.11	0.16	-
Ba	6.71	9.59	-
Ni	0.04	0.04	33.96
Pb	2.00	2.86	0.05
Sb	0.87	1.24	0.14
Se	7.73	11.04	0.04

* Negligible

As stated earlier, the copper concentrate input and the extraction process can profoundly affect the composition and properties of the copper anode slime. Accordingly, a comparison has been made between the bottom-cell slimes of the two different refinery plants, Sarcheshmeh bottom-cell slime, and Khatoon-Abad bottom-cell slime. Significant differences in the chemical composition of these two slimes can be observed. The copper and arsenic concentration difference between the Sarcheshmeh bottom-cell slime and the Khatoon-Abad bottom-cell slime is attributed to the dissolution of the water-soluble constituent of the Sarcheshmeh bottom-cell slime. The concentration is about 2-3 times lower in Khatoon-Abad bottom-cell slime. On the contrary, barium, lead, silver, and selenium content of the Sarcheshmeh bottom-cell slime is about 2-3 times higher than the Khatoon-Abad bottom-cell slime. The significantly higher selenium content (higher ratio of selenium to copper concentration) of the Sarcheshmeh slime is important due to the fact that refractory selenide phases of various elements, such as copper and silver can be formed and affect dissolution process. The concentration of the insoluble content of the slime such as barium, lead, silver, and selenium are concentrated 2-3 times when the initial slime lost its copper and arsenic content. In contrast with sliver, the gold content of the Sarcheshmeh bottom-cell slime was not increased following the 2-3 times increment in the concentration ratio. The difference is most probably due to the lower gold content of the Sarcheshmeh feed concentrate than the Khatoon-Abad feed concentrate. The chemical composition of the Sarcheshmeh and Khatoon-Abad copper concentrates are shown in Table 3. These significant differences can profoundly affect the slimes responses to various treatments.

3. 3. XRD Results The XRD pattern with phase composition of Sarcheshmeh bottom-cell slime, Sarcheshmeh adherent slime, and Khatoon-Abad bottom-cell slime is shown in Figure 3. According to this figure, Sarcheshmeh adherent slime mainly consists of lead sulfate, copper selenide, and mixed compounds of copper, silver, and selenium. In Sarcheshmeh bottom-cell slime, however, $BaSO_4$, $PbSO_4$, $Cu_{1.8}Se$, $Cu_4As_2Se_5$, CuSe, and Ag_2Se were detected as the main phases. There are noticeable differences between the phase composition of Sarcheshmeh adherent slime and Sarcheshmeh bottom-cell slime. $PbSO_4$ and copper selenide are the only common compounds found in both of these two slimes. In the Sarcheshmeh bottom-cell slime, various compounds of selenium are observed. This is due to the different chemical composition of the slimes. In the XRD pattern of the Khatoon-Abad bottom-cell slime, dominant peaks of barium sulfate and silver selenide were observed. In addition of these two compounds, two mixed compounds of Cu, Ag and Se were also detected. Due to expressively different chemical compositions, different major phases were observed in Sarcheshmeh and Khatoon-Abad bottom-cell slimes. Sarcheshmeh bottom-cell slime contains significantly higher amounts of selenium. Accordingly, most of the elements were found in close association with Se. Selenium presented in all of the detected phases in the XRD pattern, except lead sulfate. On the other hand, among the detected compounds at Khatoon-Abad bottom-cell slime, only two of them has contained selenium.

TABLE 3. Chemical composition of Sarcheshmeh and Khatoon-Abad copper concentrate

Element	Sarcheshmeh concentrate	Khatoon-Abad concentrate
Cu	26-30 %	30-35 %
Fe	25-26	21-22 %
As	0.02 %	0.03 %
Sb	0.03 %	0.01 %
Pb	0.23 %	0.01 %
Ag	50 ppm	40 ppm
Au	1.3 ppm	1.4 ppm
Bi	160 ppm	96 ppm
Se	140 ppm	80 ppm
Te	42 ppm	34 ppm

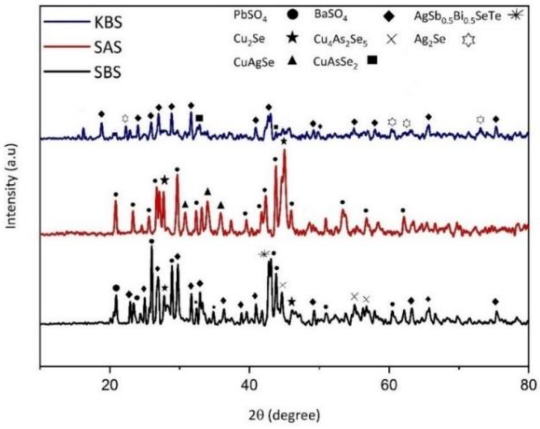

Figure 3. XRD patterns and phase detection of Sarcheshmeh adherent slime (SAS), Sarcheshmeh bottom-cell slime (SBS), and Khatoon-Abad bottom-cell slime (KBS)

3. 3. SEM Results The morphology and the chemical composition of various phases in the slimes were studied using SEM-EDS analysis. The results are shown in Figure 4. SEM image of Sarcheshmeh adherent slime is shown in Figure 4(c). Various phase compositions with a particle size below 10 µm are apparent in this figure. For phase detection, EDS analysis was performed for three different points on the SEM image. Oxide matrix phases consisting of Cu, Ag, Se, Pb, and as were identified in all of the three plotted points. However, the concentration of these elements varied with the morphology of the phases. At point 1, a spherical phase with high silver content was observed. Point 2 had a similar chemical composition to point 1; however, its sulfur content was relatively higher. The morphology of the detected phase at point 2 was different from the one found at point 1. Phase morphology at point 2 was more like drawn-out plates. A multifaceted phase with high copper and selenium content was identified at point 3. Tellurium and selenium content at point 3 was much higher in comparison to point 1 and 2, which indicates that Te is probably associated with the selenide phases.

The SEM result of Sarcheshmeh bottom-cell slime and EDS map of Cu, Se, Te, Ag, and Sb are shown in Figure 4(a). The chemical composition of the phases with different morphologies was studied using EDS analysis. Particles of this slime seemed to be agglomerated in some areas. Four different points on the SEM image of the Sarcheshmeh bottom-cell slime with various morphologies and color tones were selected for EDS analysis. At point 1, Sb, Te, and oxygen are presented with the highest concentration, which indicates the formation of oxide complex phases of these elements. Point 2 seems to be a representation of lead sulfate, which is also in a lighter color (compounds with higher masses are observed in lighter colors in SEM backscattered images). Points 3 and 4 consists of Ag, Se, and Cu with minor amounts of Te. Another finding regarding points 3 and 4, in which the highest copper concentrations were observed, is that the Ba concentration at these points are relatively high (18 to 25%). Accordingly, copper recovery can be affected by its association with barium sulfate and the possible agglomerated Ba-Cu particles. Barium sulfate does not dissolve in acidic medium. The distribution of the Cu, Se, Te, Ag, and Sb are also shown in the same figure. It can be seen that some elements have a similar distribution pattern. Cu is presented within the same area of Se, which suggests the presence of copper selenide compounds. Te and Sb have similar distribution patterns. Te and Sb zones are surrounded by Cu-Se areas. Accordingly, Te and Sb are majorly present at the core distribution of copper selenide compounds. The SEM image of Khatoon-Abad bottom-cell slime is shown in Figure 4(b). In this figure, phases with spherical, semicircular, elliptical, and drawn-plate shapes were observed. In total, seven points on the SEM image with different morphologies were analyzed using EDS. Point 1 was determined to be barium sulfate with the morphology of spherical-shape. The particle size of this compound was found to be larger in comparison to other phases. Unlike Sarcheshmeh bottom cell slime, barium particles were only found in larger particle size and were not in association with other elements. The other six remaining points were copper selenide phases carrying different elements, such as silver, arsenic, and tellurium. According to the previous studies, these phases could be Cu_2Se, Ag_2Se, AgCuSe, AgCu(Se,Te), and $(Cu,Ag)_2Se$. These phases are visible in spherical and semicircular, elliptical, and drawn-out plate shapes. The EDS map of Khatoon-Abad bottom-cell slime is also shown in this figure. According to this figure, copper can be seen to have a uniform distribution throughout the studied area. Similar to Sarcheshmeh bottom-cell slime, tellurium was found to be in close association with Sb and Se. Ag particles were also found in association with corresponding selenium compounds.

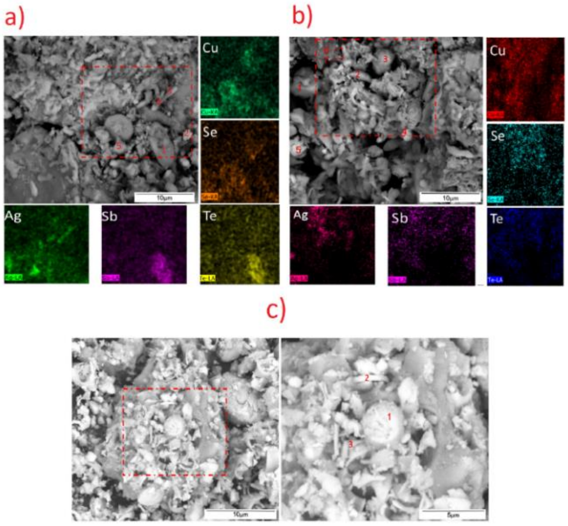

Figure 4. SEM image of (a) Sarcheshmeh bottom-cell slime (SBS), (b) Khatoon-Abad bottom-cell slime (KBS) and (c) Sarcheshmeh adherent slime (SAS)

By comparing the SEM results of Sarcheshmeh bottom-cell slime and Khatoon-Abad bottom-cell slime, it can be seen that the phase morphologies were almost similar in both slimes. Phases with the spherical, drawn-out plate, and semicircular shapes were majorly observed in both cases. However, the chemical compositions of the minor phases were different. Because of the high copper content of the Khatoon-Abad bottom-cell slime, most of the phases were detected in association with copper. In the case of Sarcheshmeh bottom-cell slime, a similar case was observed for selenium.

3. 4. Particle Size Results

Screen analysis was performed to determine the particle size of the Sarcheshmeh bottom-cell slime and Khatoon-Abad bottom-cell slime, and also, to determine the distribution of precious metals at different particle sizes within these slimes. The cumulative particle size distribution of both slimes is shown in Figure 5. According to this figure, the particle size of Sarcheshmeh bottom-cell slime is relatively smaller than Khatoon-Abad bottom-cell slime. The reason for the particle size difference could be the difference of the injected barium sulfate particle size during casting, the agglomeration of the particles, and the difference in the anode solidification time. The longer the solidification time, the larger the particle size. 80% passing particle size of the Sarcheshmeh bottom-cell slime was below 38 microns, whereas the P80 for the Khatoon-Abad slime was measured around 106 microns.

It has been shown that barium particles are mostly agglomerated at particle sizes above 17 microns. Accordingly, the possibility of the physical separation of the barium sulfate particles from the rest of the slime using the hydrocyclone or the sieve was investigated. As

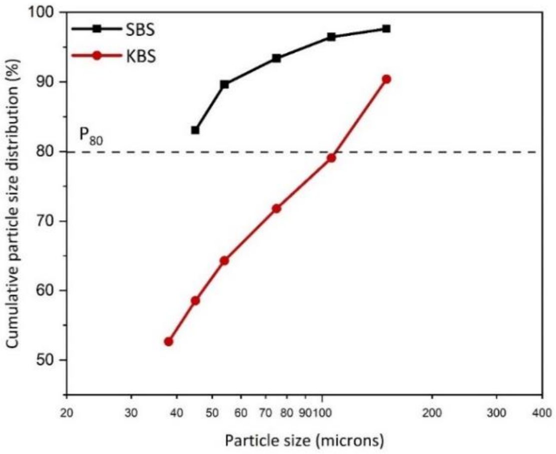

Figure 5. Particle distribution of Sarcheshmeh bottom-cell slime (SBS) and Khatoon-Abad bottom-cell slime (KBS)

stated earlier, the presence of barium can impose challenges in recovery of precious metals. The high abrasion of the barium sulfate can cause the decopperrizing tank to get corroded very fast. A shaking screen can help to separate the unwanted particles from the rest of the slime. The chemical composition of the slimes, with particle size above 38 microns, was determined using the ICP-MS. The results are summarized in Table 4. According to this table, due to the presence of a significant fraction of precious metals in particles above 38 microns, physical separation of barium through sieve analysis does not seem practical.

3. 5. STA Results Simultaneous thermal analysis of TGA and DTA was carried out to determine the interaction of the Sarcheshmeh bottom-cell slime and Khatoon-Abad bottom-cell slime compounds with temperature. Among the known recycling process, a pyrometallurgical pretreatment of the slime is popular. Oxidative roasting, sulfation roasting, and soda roasting are the industrial examples of the few pretreatment processes. As a result, the TGA and DTA results are essential in the design of the slime recovery processes. The results are shown in Figure 6. According to this figure, the TG curve of the Sarcheshmeh bottom-cell slime (Figure 6(a)) can be divided into three main parts: First is the initial mass loss, that happens at temperatures below 400˚C, second is the simultaneous mass loss and mass gain that happens from 400ºC to around 600ºC, and third is the final mass loss which happened at temperatures above 600ºC. Sarcheshmeh bottom-cell slime contains considerable amount of Se. Accordingly, most of the thermal interactions are based on selenium bearing compounds.

The initial mass loss at temperatures below 400ºC originates from removing moisture, chemically bonded water, and evaporation of volatile compounds. The free selenium and silver selenide are the main volatile compounds. Evaporation of selenium compounds may occur as following [18]:

TABLE 4. Distribution of Sarcheshmeh bottom-cell slime and Khatoon-Abad bottom-cell slime components in particle sizes above 38 microns

	Sarcheshmeh bottom-cell slime		Khatoon-Abad bottom-cell slime	
Element	**Wt. % in fraction >38 microns**	**Fraction presented at >38 microns**	**Wt. % in fraction >38 microns**	**Fraction presented at >38 microns**
Ba	30.17	23.39	9.34	47.70
Cu	4.36	8.79	25.88	57.02
Au	0.08	8.22	0.118	58.95
Ag	3.28	8.15	5.32	5.23
Se	8.19	3.98	11.80	42.65
Te	0.21	51.93	0.56	86.76

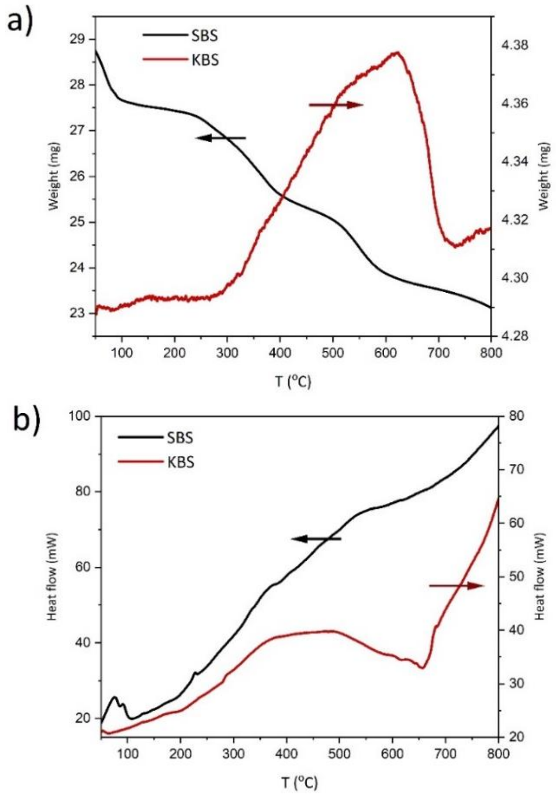

Figure 6. Thermal analysis results of Sarcheshmeh bottom-cell slime (SBS) and Khatoon-Abad bottom-cell slime (KBS) a) TGA results b) DTA results

$Se_{(s)} = Se_{(g)}$ $227.5<\Delta H\ (kJ)<229.9$ (1)

$Se_{(s)} + O_{2(g)} = SeO_{2(g)}$ $-128.8<\Delta H\ (kJ)<-118.9$ (2)

$Ag_2Se_{(s)} = 2Ag_{(s)} + Se_{(g)}$ $260.0<\Delta H\ (kJ)<269.3$ (3)

$Ag_2Se_{(s)} + O_{2(g)} = 2Ag_{(s)} + SeO_{2(g)}$ $-81.6<\Delta H\ (kJ)<-79.9$ (4)

The standard Gibb's free energy of these reactions are shown in Figure 7. Some of the above-stated reactions have positive standard Gibb's free energy, which imputes that they cannot occur in thermodynamic standard condition. They can however occur in non-standard conditions. Three endothermic peaks have been observed in the temperature range of 100 to 400°C (Figure 6(b)). The first two endothermic peaks have occurred at temperatures below 100ºC, which is related to the evaporation of the sample's moisture. Another endothermic peak is visible at around 200ºC, which is probably for the evaporation of the elemental selenium and molecular water.

After 300^{o}C, a mass gain happens by the reactions between anode slime components, the gas products, and the oxidizing atmosphere. The formation of silver selenite can happen according to the following reaction:

$$Ag_2Se_{(s)} + 1.5O_{2(g)} = Ag_2SeO_{3(s)} \quad -288.2<\Delta H\ (kJ)<-239.2 \quad (5)$$

Moreover, copper compounds, such as Cu_2Se and Cu_2O, can also react with oxygen and sulfur dioxide to form solids with higher weights [19]:

$$Cu_2Se_{(s)} + 2O_{2(g)} = Cu_2SeO_{4(s)} \quad -288.2<\Delta H\ (kJ)<-239.2 \quad (6)$$

$$Cu_2O_{(s)} + 2SO_2 + 1.5O_{2(g)} = CuSO_{4(s)} \quad -288.2<\Delta H\ (kJ)<-239.2 \quad (7)$$

Based on reaction 7, for the conversion of copper oxide to copper sulfate, the presence of sulfur dioxide gas is essential. Considering a low concentration of elemental sulfur in slime, the sulfur dioxide can be provided from decomposition of other sulfide compounds. Due to the high selenium content of the Sarcheshmeh slime, the two above reactions possibly happen at lesser quantities in comparison to selenium compounds reactions. The formation of these compounds could result in a slight mass gain of the slime. As the temperature increases, the previous mass loss reaction, due to the endothermic nature of the reactions, happens at higher rates. In the case of Sarcheshmeh bottom-cell slime, the mass loss reactions have overcome mass gain reactions, which eventually resulted in an overall mass loss. Finally, the mass losses at temperatures above 600^{o}C can represent the decomposition of silver selenide and sulfated compounds, such as $BaSO_4$, $PbSO_4$, and $CuSO_4$ through the following reactions [19-22]:

$$Ag_2SeO_{3(s)} + SO_{3(g)} = Ag_2SO_{4(s)} + SeO_{2(g)} \quad -106.1<\Delta H\ (kJ)<-99.5 \quad (8)$$

$$PbSO_{4(s)} = PbO_{(s)} + SO_{3(g)} \quad 292.0<\Delta H\ (kJ)<299.9 \quad (9)$$

$$BaSO_{4(s)} = BaO_{(s)} + SO_{2(g)} + 0.5O_{2(g)} \quad 562.2<\Delta H\ (kJ)<603.3 \quad (10)$$

$$CuSO_{4(s)} = CuO_{(s)} + SO_{2(g)} + 0.5O_{2(g)} \quad 303.7<\Delta H\ (kJ)<308.9 \quad (11)$$

A significantly different behavior was observed in Khatoon-Abad bottom-cell slime thermal peaks comparing to the Sarcheshmeh bottom-cell slime. Khatoon-Abad bottom-cell slime was dried previously at 90^{o}C for 24 hours. Accordingly, no weight loss or endothermic peaks were observed pre-100^{o}C. The Khatoon-Abad bottom-cell slime contains a significant amount of copper; therefore, most of the interaction can be attributed to the copper-containing compounds. Till 300^{o}C, no significant interaction is evident in the thermograph. However, after 300^{o}C, up to around 600^{o}C, a considerable mass gain was observed in the TGA graph (Figure 6(a)). The mass gain can be attributed to the oxidation of different copper compounds. The mass loss reaction may also occur similar to the reactions previously discussed. Their effect, however, could be potentially masked by mass gain reactions. A mass gain as the result of oxidation of copper selenide and cuprous oxide is viable according to reactions 6 and 7. Another possible reaction involving copper that could result in increased weight of the sample is as follows:

$$Cu_2S_{(s)} + 7.5O_{2(g)} = 2CuSO_{4(s)} + 3Cu_2O + 2SO_2 \quad -288.2<\Delta H\ (kJ)<-239.2 \quad (12)$$

The combination of reaction 12 with reactions 6 and 7 can cause an exothermic peak at around 600^{o}C. After 600^{o}C, due to the decomposition of sulfates of different elements, such as copper, barium, and lead, a mass loss has happened. Due to the differences in the chemical composition, especially in copper and selenium concentrations, distinct behaviors were observed in the interaction of these slimes with temperature. A consistent mass loss was observed in the TGA result of Sarcheshmeh bottom-cell slime with increasing temperature as a result of high selenium content. An opposite result was observed in the Khatoon-Abad bottom-cell slime TGA graph. A consistent mass gain trend was observed up to temperatures around 600^{o}C, due to the oxidation of copper-containing compounds.

As stated earlier, pyrometallurgical pretreatments are common in copper anode slime processing. Accordingly, besides the above explanations, a brief thermodynamic assessment of the discussed reactions are presented below. Since the thermal interactions in the studied slimes (Sarcheshmeh and Khatoon-Abad bottom-cell slimes) were mostly found in association with Cu-Se-Ag compounds, oxidative roasting in the systems of Ag-Se-O and Cu-Se-O has been studied.

In Figures 7 and 8 the standard Gibbs free energy (ΔG^0) of Ag-Se-O and Cu-Se-O system and their dominant species at various temperature and oxygen pressure are illustrated. The thermodynamic data was obtained from HSC software database (Version 6, Outokumpu research). It should be noted that the data presented in these figures are ΔG^0 not ΔG. Accordingly, although some reactions have positive ΔG^0, they could still thermodynamically happen if their ΔG becomes negative in a non-standard condition. Oxidizing roasting is mostly applied to convert elements, such as copper, nickel, and tellurium into soluble compounds in sulfuric acid solution and also evaporate the selenium. According to Figure 7, evaporation of selenium is mostly happening by conversion of Se-containing compounds to $SeO_{2\ (g)}$. The process of Se removal involves the formation of intermediate selenite species. As discussed in the previous section, in bottom-cell copper anode slimes, selenium can present as silver selenide (Ag_2Se). At lower temperatures (around <400°C), due to relatively lower ΔG^0, the transformation of Ag_2Se to either intermediate species, such as Ag_2SeO_3 or directly to SeO_2 happens. Intermediate species of silver and selenium tend to become less stable with increasing temperature, which leads to the evaporation of selenium as SeO_2. This is also evident in the speciation diagram of Ag-Se-O (Figure 7(b)). At temperatures above 300°C and oxygen partial pressure of 10^{-5}, SeO_2 is mostly stable.

Similar to Ag-Se-O system, the same graphs were plotted for Cu-Se-O system. The presence of copper selenide is possible in bottom-cell anode slimes. The process of selenium removal and transformation of copper selenide to soluble copper species is evident in Figure 8. At temperatures below 600°C, the transformation of Cu_2Se to copper selenate happens spontaneously. By increasing temperature, the Gibbs free energy of the decomposition reaction of copper selenate to CuO and SeO_2 becomes less negative. Accordingly, the thermodynamic driving force for the copper selenate decomposition becomes larger. As a result of this process, selenium can be removed from the slime as SeO_2, and also copper transforms to non-sulfide/soluble species, such as $CuSO_4$ and CuO.

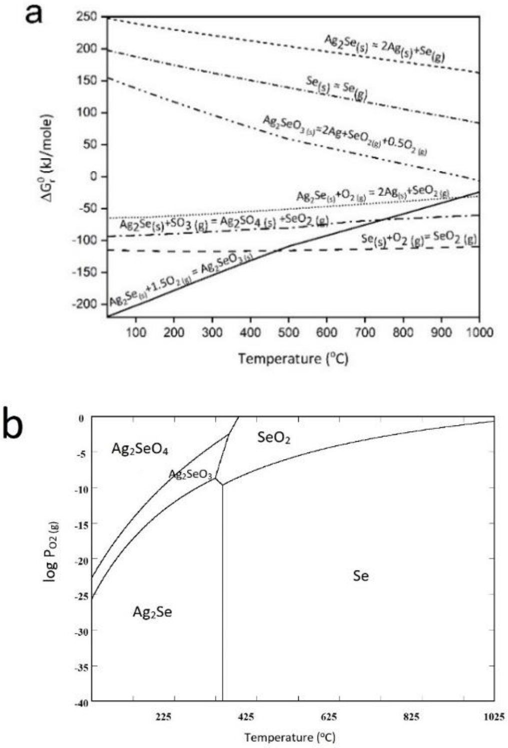

Figure 7. Oxidative roasting in Ag-Se-O system a) Standard Gibbs free energy of reaction as the function of temperature b) Effect oxygen partial pressure and temperature on Ag-Se-O species

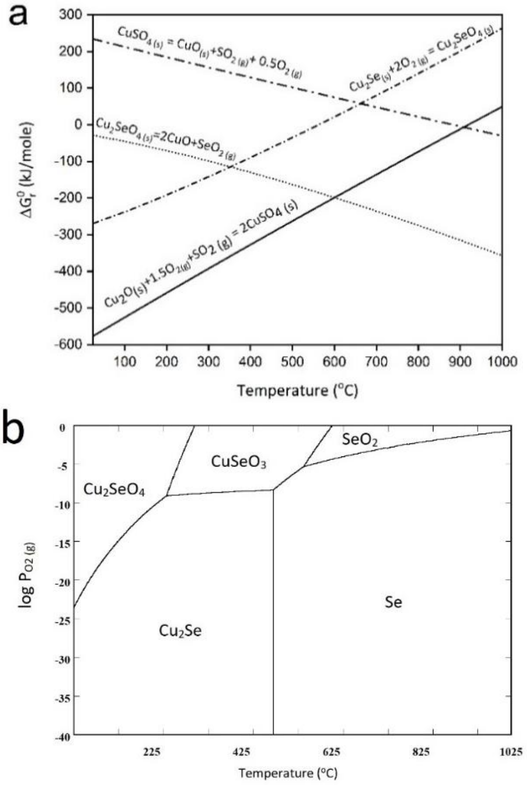

Figure 8. Oxidative roasting in Cu-Se-O system a) Standard Gibbs free energy of reaction as the function of temperature b) Effect oxygen partial pressure and temperature on Cu-Se-O species

3. 6. Decopperization

The proper characterization can strongly influence the selection of the proper method in recovery of the valuable content of the slime. In this section, as an example, a hydrometallurgical recovery of copper from Sarcheshmeh and Khatoon-Abad bottom-cell slimes, considering the characterization results, are investigated. In hydrometallurgical recovery of copper anode slime components, the first step is often the decopperization process .

Chemical analysis showed that copper concentration in Khatoon-Abad slime was considerably higher than Sarcheshmeh slime. However, a significant fraction of copper in Khatoon-Abad slime was in a form of water-soluble copper compounds. Phase composition analysis of both slimes revealed that copper was mostly associated with selenium, which necessities using of the oxidants for efficient copper recovery. However, the ratio of selenium to copper in Sarcheshmeh slime was significantly higher than that of it in Khatoon-Abad slime, which imputes that copper concentration of non-selenide compounds are potentially higher at Khatoon-Abad slime. This could result in easier copper dissolution. Moreover, copper selenide in Sarcheshmeh slime was agglomerated within barium sulfate, which could negatively affect the copper dissolution, especially in sulfuric acid medium.

Considering the small particle size of the Sarcheshmeh slime, further grinding may not be feasible in liberating of the copper particles from barium sulfate. Accordingly, conventional leaching may not be a suitable solution for the full copper recovery of Sarcheshmeh slime and therefore more severe conditions in terms of temperature and pressure may be essential. Therefore, copper recovery from Sarcheshmeh and Khatoon-Abad bottom-cell slimes using sulfuric acid in a concentration similar to copper electrolyte (160 g/L) in the presence of air and oxygen as an oxidant were investigated. The copper recovery results are shown in Figure 9.

As it is evident, for recovering copper from both slimes, the presence of oxidant is required. Copper recovery was improved by substituting air with oxygen. However, considering different characteristics of the slimes, significantly different copper dissolution behavior was observed. After 12 hours, copper dissolution from Khatoon-Abad slime reached around 90% recovery, while copper recovery from Sarcheshmeh slime was limited to around 40%. As stated earlier, one possible reason for low copper dissolution of Sarcheshmeh slime can be attributed to higher selenium to copper ratio and agglomeration of copper particles in barium sulfate. Therefore, higher copper dissolution at the elevated pressures and temperatures is predicted. Accordingly, another experiment in autoclave was conducted over the Sarcheshmeh slime. The temperature and pressure in this experiment were 120°C and 8 bars, respectively. The pressure was adjusted by oxygen gas. The obtained results are shown in Figure 10.

As it is evident, copper recovery was significantly improved by increasing temperature and pressure. Increasing pressure increases the dissolved oxygen content of the leachate and increasing temperature helps the dissolution reaction to occur faster; both resulted in higher copper dissolution. Obviously, a difference in the phase composition of the slimes has caused different decopperization results. For Khatoon-Abad slime, an atmospheric leaching was efficient; whereas for Sarcheshmeh slime, leaching in autoclave was requiered.

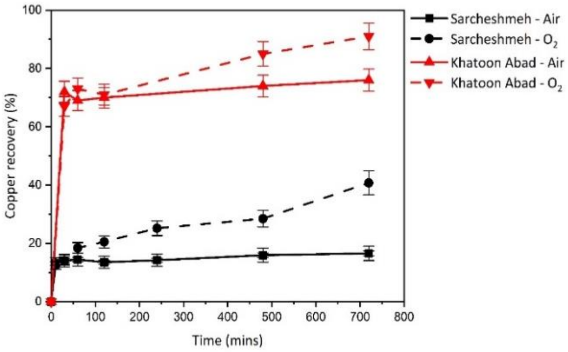

Figure 9. Copper dissolution behavior from Sarcheshmeh and Khatoon-Abad bottom-cell slimes

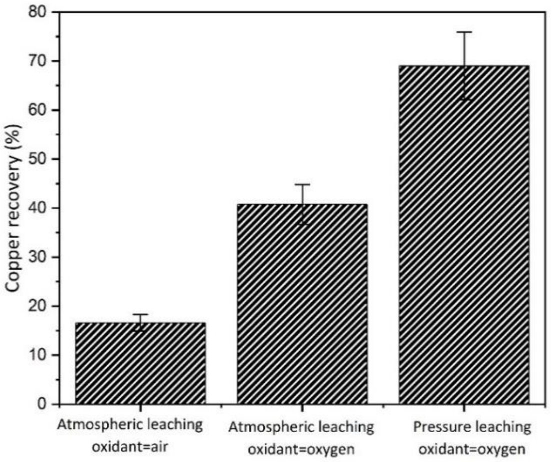

Figure 10. Effect of leaching method on copper dissolution from Sarcheshmeh bottom-cell slime

This difference will also extend to the recovery of other constituent elements of the slime such as tellurium and selenium .

4. CONCLUSION

In this research, two different types of slimes from Sarcheshmeh copper complex and Khatoon-Abad copper complex were characterized and compared. The results showed that characteristic properties of the slime could significantly vary from one refinery plant to another due to the differences in feed material and smelting production processes. Chemical composition of the slimes can result in profound differences in the major phase constituents, the minor phases, particle size, and interaction of slimes with temperature. Sarcheshmeh bottom-cell slime contained high amounts of selenium; therefore, most of the other slime components were observed in association with this element. Khatoon-Abad bottom-cell slime, however, contained significant amounts of copper. Accordingly, other components were closely linked to copper. These distinctions resulted in significant differences in TGA-DTA results. Since Sarcheshmeh bottom-cell slime contained a high amount of selenium, most of thermal interactions were with Se-containing compounds. In the case of Khatoon-Abad bottom-cell slime, however, due to high copper content, most of thermal interactions were attributed to the Cu-containing compounds. Adherent slime of Sarcheshmeh refinery plant was also characterized and compared to Sarcheshmeh bottom-cell slime. The adherent slime was found free of barium sulfate. The variance in characteristic properties of the slimes have resulted in significant differences in copper dissolution behavior of slime. Due to the association of copper with selenium and barium in the Sarcheshmeh slime, copper content of the slime was only effectively dissolved at elevated temperature and pressure, while high copper recovery from Khatoon-Abad slime was obtained at moderate conditions.

5. CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

6. ACKNOWLEDGMENTS

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Persian Abstract

چکیده

لجن آندی مس، محصول جانبی با ارزشی است که در طول فرایند تصفیه الکتریکی مس تولید می شود و یکی از مهمترین منابع ثانویه بازیابی فلزات طلا، نقره، سلنیوم، تلوریوم، مس و فلزات گروه پلاتین می باشد. مشخصه یابی کامل لجن آندی پیش نیاز بازیابی مطلوب این فلزات می باشد. در این تحقیق، مشخصه یابی کامل لجن آندی مجتمع مس سرچشمه و مجتمع مس خاتون آباد با استفاده از روشهای ICP، XRD، XRF، SEM، EDS، TGA، DTA و تعیین اندازه دانه صورت پذیرفت. علاوه بر لجن جمع آوری شده در کف سلول، مشخصات لجن چسبنده به سطح آند در مجتمع مس سرچشمه نیز مورد بررسی و مقایسه قرار گرفت. مشاهدات نشانگر اندازه بسیار کوچکتر لجن سرچشمه نسبت به لجن خاتون آباد می باشد. در عین حال، قسمت عمده طلا و تلوریوم در هر دو لجن، در ذرات درشت تر از ۳۸ میکرون تجمع یافته است. تفاوت عمده در مشخصات حرارتی دو لجن نیز متاثر از میزان سلنیوم محتوی دو لجن می باشد. در لجن خاتون آباد، غلظتهای بالاتر ترکیبات مس دار غیر سلنیدی، انحلال بالاتر مس محتوی لجن را به همراه داشته است. اختلاف چشمگیر در انحلال مس محتوی این دو لجن بواسطه حضور فازهای سلنیدی مس و محصور شدن مس در ذرات کلوخه ای سولفات باریم است. با توجه به تفاوتهای فوق، انحلال اتمسفری محتوی مس لجن خاتون آباد برابر ۹۰ درصد و در لجن سرچشمه برابر ٤۰ درصد مشاهده گردید.