











# Analysis of the generation and physical-chemical characteristics of slag from the recycling of lead-acid batteries

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## Abstract

The lead produced in Brazil comes entirely from the lead-acid battery recycling industry. During the process, 100 to 350 kg of slag is generated for each ton of metallic lead produced. This large amount of waste generated causes serious environmental problems, where reuse and reducing toxicity are measures to resolve its negative impact. Thus, the work methodology adopted was a bibliographic survey addressing the battery recycling process (pyrometallurgy), presenting data, and discussing the toxicity of slag, its generation, its physical and chemical characteristics, and the possible waste environmental impact if not treated and disposed of properly. The results show that lead slag contains some toxic elements, such as lead, zinc, and cadmium, and that if it is not disposed of properly, it will become a paramount environmental problem. Lead slag contains large amounts of silicon, calcium, iron, copper, and other value-added metals, which can be reused as secondary resources. Thus, improper disposal of lead waste can cause critical waste of resources. This article highlights the problems that need to be faced to guarantee the industry's sustainable development, seeking to reduce the use of inputs and natural resources and minimize environmental problems.

**Keywords:** Lead-acid batteries; Solid waste; Lead refining slag.

## 1 Introduction

Recycling lead from waste lead-acid batteries generates high amounts of residue during the melting and reduction stages, in which the lead is recovered in its metallic form and the impurities present in the raw material, along with other materials added to promote the reactions necessary for the process, form the slag. During the process it is estimated that for each ton of metallic lead produced, 100 kg to 350 kg of slag is generated. Another estimate indicates that the annual production value of waste is around 4 million tons worldwide [1].

In Brazil, the production of lead comes from recycling lead-acid battery waste, and the slag is classified as hazardous

waste, according to Brazilian standard NBR 10004:2004 of the Brazilian Association of Technical Standards (ABNT), and in the absence of more appropriate options, the final destination is the disposal of slag in landfills for hazardous industrial waste [2,3].

The objective of this work was, first, to approach the pyrometallurgical process, as well as the generation and characteristics of lead slag, and the possible environmental impacts caused by the residue. This work is presented as a research methodology, a critical review of the literature for the central theme, adopting evaluation and critical analysis on the data obtained.

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The results show that lead slag contains some toxic elements, such as lead, zinc, and cadmium, which, if not disposed of properly, could represent a serious problem for the environment. In addition, lead slag contains large amounts of silicon, calcium, iron, copper and other valuable metals, that can be reused as secondary resources. Under these circumstances the improper disposal of lead slag can result in a critical waste of resources. Therefore, the main problem that needs to be solved to ensure the sustainable development of the lead smelting industry, is the reduction of waste generated.

## 2 Literature review

### 2.1 The process of lead production from battery recycling waste

The process of lead production from waste batteries can be described as a succession of steps, as presented in the simplified general flowchart in Figure 1.

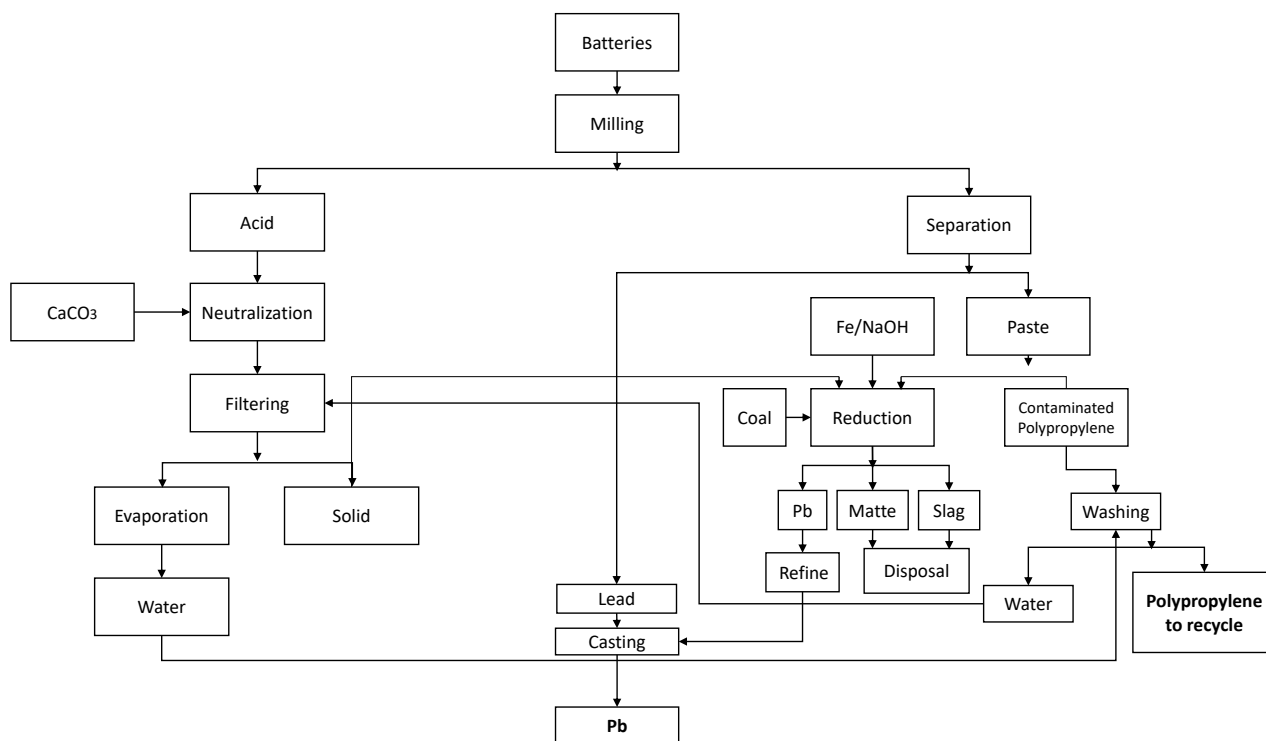
According to Gomes et al. [1], the recycling process begins with a mechanical processing that essentially involves unit operations that aims to physically separate or concentrate materials from battery scraps. The batteries are fragmented in hammer mills, then the material goes to a gravimetric separation process (sink/float). This hydrodynamic step is used to separate the lead from the separators and the casing material in two different streams. The housing material is polypropylene (PP) and the separation can be accomplished

by the difference in density between the materials. Since the density of PP is much lower in comparison to lead, this density separation step has high efficiency, achieving almost complete separation of the different materials. The polymers are subsequently cleaned and sent for recycling in extruders to use in new battery cases or for alternative uses. The lead and paste, a combination of  $PbO_2$ ,  $PbO$ ,  $PbSO_4$  and metallic lead from the active material, are removed from the bottom of the gravimetric separation operation and sent to storage for water removal prior to casting. A typical flow process diagram is shown in Figure 2.

According to Sohn [6] and Gupta [7], the lead slag is a byproduct of the lead production process from battery recycling, and the main method of processing this material is by the pyrometallurgical route, sintering-blast furnace and the reduction process, as shown in Figure 2, including oxidation, reduction and refining steps. In the sintering-blast furnace route  $PbS$  is oxidized in the solid state to remove sulfur and produce  $PbO$  at the same time.  $PbO$  is then reduced to metallic  $Pb$  and, at this stage, the blast furnace is charged with coke. Calcium carbonate is also added to lower the melting temperature and form a molten slag in which the iron oxides are removed.

As described earlier, lead-acid battery waste is the main source of secondary lead, accounting for over 85% of the total secondary lead [5,8].

According to Gomes et al. [1] and Sun et al. [9], currently, almost 95% of the lead-acid battery recovery plants in operation are based on pyrometallurgical methods. In the secondary lead recovery process, lead sulfate ( $PbSO_4$ ) and



**Figure 1.** General flowchart of the lead recycling from waste batteries process [4].

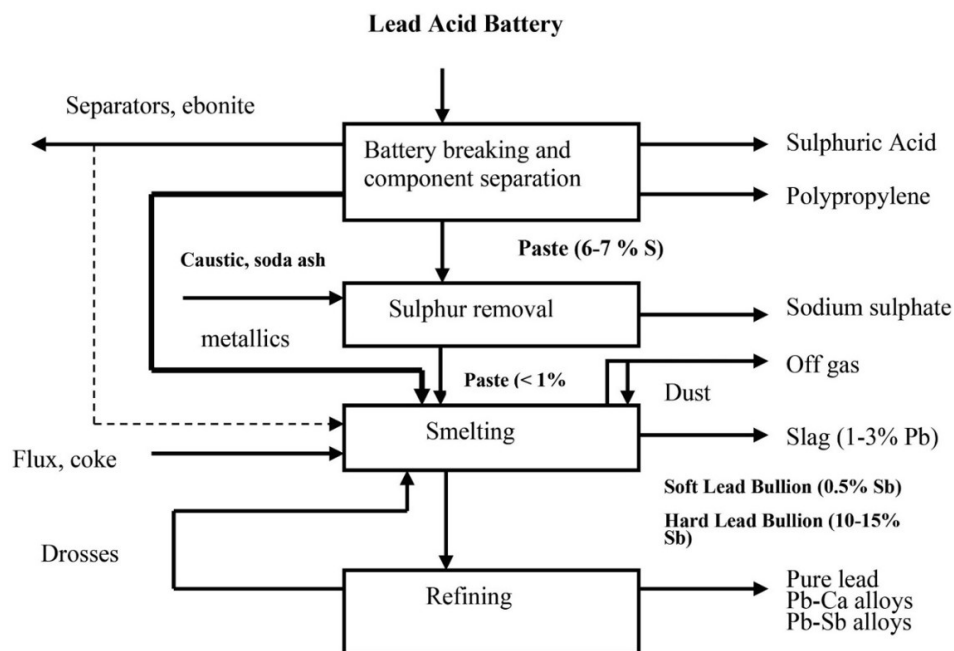


Figure 2. Flowchart of the flow process [5].

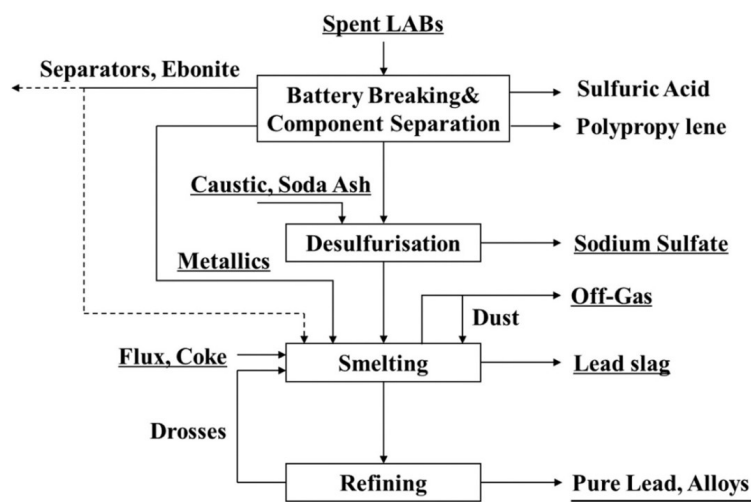
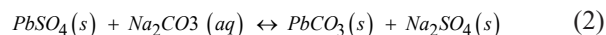
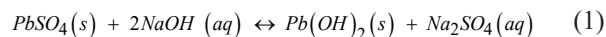


Figure 3. A typical flow chart of secondary lead recovery by indirect smelting Li et al. [10,11] and Pan et al. [12].

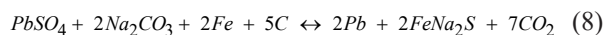
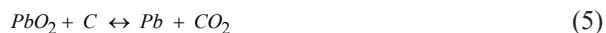
lead oxides ( $PbO$  and  $PbO_2$ ) from the waste are reduced to metallic lead ( $Pb$ ). In the traditional pyrometallurgical process, the melting temperature of lead paste in waste lead-acid batteries is more than  $1000\text{ }^\circ\text{C}$ , and sodium iron carbonate are added to this process as reducing agents.

According to Sun et al. [9], Li et al. [10,11] and Pan et al. [12], indirect smelting has good low environmental impact, and can remove most of the sulfur from the paste before smelting. A typical flow chart of secondary lead recovery in indirect smelting is shown in Figure 3. Sulfur removal is the first step in the production of refined lead. Hydrometallurgical sulfur removal is an environmentally friendly technology that transforms lead sulfate to carbonate and hydroxide. The following reactions (Equations 1 and 2)

are based on the lower solubility of  $PbCO_3$  or  $Pb(OH)_2$  when compared to  $PbSO_4$ . The insoluble  $PbCO_3$  or  $Pb(OH)_2$  collected as sludge or filter cake is then transferred to the smelter.



In the smelting process, calcium carbonate ( $CaCO_3$ ) is added as flux, while coke and iron filings are used as reducing agents; lead is then recovered from the matte by reduction with iron. The following reactions (Equations 3, 4, 5, 6, 7 and 8) are used to describe the generation of metallic lead [1,12].



## 2.2 The generation of lead slag from the battery recycling process

Throughout the casting stage of a metal, another molten phase is generated which, when cooled solidifies and forms the slag phase. Slags can perform a number of functions in casting operations, they primarily serve to remove undesirable impurities, unreduced oxides and ash generated in the combustion of solid fuel. The slag also insulates the metal from contact with the atmosphere just above the metal bath and thus prevents or delays the transfer of gaseous species. Since a molten oxide has a much lower thermal conductivity than liquid metal, the slag acts as a thermal barrier that decreases heat loss at the surface of the metal. However, slag also delays heat transfer to the metal when heat is supplied by the combustion that occurs just above the bath [3,6,13].

According to Gomes [14], slag must possess certain fundamental properties to perform the desired functions cited above, such as Table 1 below.

According to Sun et al. [5,9] and Gomes [13,14], slag is usually composed of a solution formed by different oxides and silicates, and is generated via endothermic reactions. Thus, increasing the volume of slag, increases the heat added and also increases the cost related to fuels. This higher volume of slag also increases the risk of metal insulation, decreasing the yield of metal formation and the rate of heat transfer from the furnace to the molten metal. The metal-slag interface area is important to the reaction rate, since the rate of product formation is related to the diffusion of species from the metal to the interface and the transfer of species across the interface. In consequence,

the viscosity and interfacial energy between slag and metal become parameters of great importance.

Some compounds present in liquid slag are ionic, that are usually formed of oxides, that when molten are present as cations and anions. In liquid compounds containing molten materials, elements such as Ca, Fe, K, Li, Mg, Mn, Na, Pb and Zn, can ionize, and function as bases by transferring electrons to oxygen or other anions [15].

According to Li et al. [10], slag can be basic, acidic or neutral. Due to the fact that they have basic oxides in most of their composition, the slag normally has an excess of  $O^{2-}$  ions, as the metal that forms the oxide ionizes and donates oxygen to the slag. Acid slags, on the other hand, contain an excess of acidic oxides, such as  $SiO_2$ ,  $P_2O_5$  or  $Al_2O_3$ , which normally produce complex cations by reacting with oxygen anions.

According to Li et al. [10,11] and Pan et al. [12], slag can also be oxidizing or reducing in relation to the molten metal. An oxidizing slag transfers oxygen to the melt, promoting an oxidation. To achieve this, metallic oxides can be added, which dissociate at the metal-slag interface and release oxygen to the melt. For the reducing slag, however, this oxygen transfer does not occur. The reducing condition is satisfied by the addition of carbon that acts as a reducing agent. An important control variable of the process involving metal-slag reactions is the composition of the slag generated, which in turn is managed by a number of factors, such as added fluxes, the reductant used, combustion gases, among others. Assuming that the function of the slag is to accept the non-desirable components and leave the molten metal in its purest state, the activity of these components treated as impurities must be as low as possible, which makes them better accepted by the slag phase, preventing the occurrence of the reverse reaction.

## 2.3 Physical and chemical characteristics of lead slag

According to Gomes [3], the slag is usually rich in iron, most often in the form of oxides, but its composition can vary according to the process and the type of raw material used. Elemental analysis data for the slag are presented in Table 2.

According to Pan et al. [12], the composition of lead slag usually falls within the following ranges, as shown in Table 3.

**Table 1.** Fundamental properties of slag for performance of desired functions

Fundamental properties of a lead slag	
a.	A lower melting point than the liquid metal to maintain a high fluidity liquid slag, covering the metal surface completely and allowing good contact with the metal;
b.	A lower density than, and immiscibility to, the liquid metal, remaining just above the melt as a distinct liquid layer and consequently being able to accept impurities that are generally lighter than the metal;
c.	The correct composition to be able to accept and react with impurities and be immiscible to the metal. This property is directly related to the addition of fluxing and slag materials in the charge.

**Source:** Adapted from Sun et al. [9] and Gomes [14].

**Table 2.** Elemental analysis for the slag produced in a rotary kiln

Element	Arithmetic mass (% by mass)	Range (% by mass)
S	16.82	13.3 - 19.7
Na	22.39	16.5 - 25.7
Fe	19.96	16.8 - 25.7
Pb	2.55	0.66 - 7.53
Zn	0.64	0.01 - 1.22
Cd	0.05	0.01 - 0.08
Cu	0.7	0.37 - 1.06
Ni	0.03	0.01 - 0.10
Mn	0.16	0.14 - 0.24
Ca	1.2	0.78 - 1.67
Mg	0.15	0.05 - 0.25
Al	0.35	0.16 - 0.58
Sn	0.78	0.44 - 1.08
P	0.35	0.30 - 0.39
As	0.26	0.10 - 0.50
Sb	0.24	0.05 - 0.70
Si	2.41	1.89 - 3.02
Cl	1.5	-
<b>Total</b>	<b>70.54</b>	<b>65.28 - 74.46</b>

Source: Adapted from Gomes [14].

**Table 3.** Chemical composition range of slag

Element	Range (% by mass)
Fe	20.1 - 55.68
Ca	1.3 - 22.43
Si	2.6 - 20
Zn	0.12 - 1.10
Pb	1.15 - 21.97
Cu	0.25 - 1.30
S	0.52 - 20.50

Source: Adapted from Pan et al. [12].

Table 4 presents different slag chemical compositions.

The morphological and structural characteristics of lead slag can be determined by X-ray diffraction analysis (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The mineralogical phases of the slag are presented in Table 5.

The main compounds in the slag are iron complexes consisting of Wustite (FeO), Pyrrhotite (FeS), Magnetite (Fe<sub>3</sub>O<sub>4</sub>), and small amounts of Phayalite (Fe<sub>2</sub>SiO<sub>4</sub>). Lead is present in small amounts as Galena (PbS), Litharge (PbO) and metallic lead (Pb) phases. Sulfur is associated with iron and sodium as Pyrrhotite (FeS).

### 3 Environmental aspects

#### 3.1 The environmental impacts caused by the slag

According to Gomes et al. [1], currently the main method of disposal for the slag is the hazardous industrial

**Table 4.** Chemical composition of the slag

Element	Arithmetic mass (% by mass)
Fe	45.46
Ca	7.99
Si	7
Zn	0.58
Pb	10.14
Cu	0.95
S	10.05
Al	5.12

Source: Adapted from Pan et al. [12].

**Table 5.** Composition of the mineralogical phases of the slag

Groups	Fases	
<b>Silicates</b>	Faialite (Fe <sub>2</sub> SiO <sub>4</sub> )	
	Olivine [(Ca, Fe <sub>2</sub> ) <sub>2</sub> ]	
	Kirschsteinite (CaFeSiO <sub>4</sub> )	
	Hedenbergite (CaFeSi <sub>2</sub> O <sub>6</sub> )	
	Willemite (Zn <sub>2</sub> SiO <sub>4</sub> )	
	Melilite [Ca <sub>2</sub> (Fe, Mg, Zn, Al) (Si, Al) <sub>2</sub> O <sub>7</sub> ]	
	Espinelio (Zn, Mg, Fe) (Fe, Al, Cr) <sub>2</sub> O <sub>4</sub>	
	Franklinite (ZnFe <sub>2</sub> O <sub>4</sub> )	
	<b>Oxides</b>	Magnetite (Fe <sub>3</sub> O <sub>4</sub> )
		Wustita (FeO)
Litargirium (PbO)		
Galena (PbS)		
Pyrrhotite (Fe <sub>1-x</sub> S)		
<b>Sulfates</b>		Wurtzita (ZnS)
		Covellite (CuS)
	Anilite (Cu <sub>7</sub> S <sub>4</sub> )	
	Chalcopyrite (CuFeS <sub>2</sub> )	
	Bornita (Cu <sub>5</sub> FeS <sub>4</sub> )	
	Chalcocite (Cu <sub>2</sub> S)	
	Digenite (Cu <sub>9+x</sub> S <sub>5</sub> )	
	Cubanite (CuFe <sub>2</sub> S <sub>3</sub> )	
<b>Metals</b>	Marmatite (Zn <sub>1-x</sub> Fe <sub>x</sub> S)	
	Metallic lead (Pb)	
	Copper (Cu)	

Source: Adapted from Pan et al. [12].

waste landfills. According to Yin et al. [16], the environmental stability of lead slag depends on several factors such as altered pHs, the mineral phase in the slag, the influence of the atmosphere, and the interaction time of slag and water. Lead slag releases Zn, Fe and Ca more easily under acidic conditions, and is easily eroded with water.

The presence of oxygen increases oxidative weathering and promotes the formation of secondary phases of oxides and carbonates that can more readily release the toxic elements. The concentration of toxic elements in lead slag increases with prolonged leaching time. According to Zheng et al. [17], Zhou et al. [18] and Silva et al. [19], due to weathering and rainfall, toxic elements in the slag, such as Pd, Zn, can penetrate the soil, causing contamination if measures to prevent infiltration are not used. According

to Hilts [20] and Tukker et al. [21], as the toxic elements in the soil are not easily broken down by microorganisms, they can migrate to plants and consequently affect animal growth, in the case of pasture and other crops, and, eventually, can cause a harmful effect on human health through the food chain.

Also, according to Silva et al. [19], the toxic elements in the slag can contaminate surface and groundwater through rainfall, which can pose a serious threat to local populations, affecting the treatment and supply of drinking water. In addition, lead slag can easily turn to dust in open storage conditions. If the toxic elements from the slag enter the atmosphere, this can result in air pollution and even a threat to human health.

### 3.2 Classification of Pb slag as solid industrial waste according to ABNT NBR 10.004:2004

According to Gomes [14], in Brazil, the classification of industrial waste is based on the ABNT NBR 10.004:2004 [2] technical standard, that classifies the waste in two classes, one of which has two subclasses, as shown in Table 6 below.

According to Gomes [3,14] and ABNT NBR 10.005:2004 [2], a waste is hazardous when it presents one or more characteristics that, due to its physical, chemical or infectious properties, pose risks to public health and the environment, as shown in Table 7 below.

When toxic waste is disposed of in the soil, leaching and, consequently, groundwater pollution can occur. Toxicity is defined by tests in laboratories according to the ABNT NBR 10.005:2004 [2] standard for Leaching of Waste. From the tests, the maximum concentrations of contaminants in the leachate can be identified. Above which, it becomes harmful to human health and the environment.

In accordance with ABNT NBR 10.004.2004, Table 8 was prepared, indicating the maximum concentration of certain problematic elements in the leachate.

According to Pan et al. [12], many leaching tests are conducted to evaluate the natural wear or landfill conditions of lead slag, and the purpose of the test is to classify the lead slag as hazardous or non-hazardous materials to determine whether the lead slag can be disposed of in a landfill without special treatment, in accordance to ABNT NBR 10.005:2004 [2] and Gomes [3,14].

## 4 Conclusion

From the literature review and the discussions held, considering the stages of oxidation, reduction and refining and the high energy consumption, the pyrometallurgical process is highly efficient, relating mainly to the recovery of lead by recycling batteries, being considered a traditional and already established method in Brazil.

As for the results about the slag, presented in Tables 2, 3, 4 and 8, the concentrations and maximum

**Table 6.** Classes for Classifying Industrial Waste

Class I	Class II
Dangerous	Non-Dangerous
-	-
-	Class II A
-	Non-Intact
-	Class II B
-	Inert

**Table 7.** Characteristics of hazardous waste

Characteristics	Effects
<b>Flammability</b>	Wastes that may ignite under certain conditions or are spontaneously flammable
<b>Corrosivity</b>	Corrosive wastes are acids or bases that are capable of corroding metals
<b>Reactivity</b>	Reactive wastes are unstable under “normal” conditions. They can cause explosions, toxic fumes, gases or vapors when mixed with water
<b>Toxicity</b>	Toxic wastes are harmful or fatal when ingested or absorbed

Source: Adapted from ABNT NBR 10.005:2004 [2] and Gomes [3,14].

**Table 8.** Concentration - maximum limit in the extract obtained in the leaching test

Parameters	Maximum limit in leachate (mg/L)
	Inorganics
<b>Arsenic</b>	1
<b>Barium</b>	70
<b>Cadmium</b>	0.5
<b>Lead</b>	1
<b>Total chromium</b>	5
<b>Mercury</b>	0.1
<b>Selenium</b>	1

Source: Adapted from ABNT NBR 10.005:2004 [2] and USEPA [22].

allowable limits of lead and other metals according to ABNT NBR 10.004:2004 classify the slag as hazardous waste class I. Thus, the slag must be discarded and sent to a hazardous industrial waste landfill.

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