



Research article

Stabilization of residual mercury from gold mining as metacinnabar and cinnabar in ball mills on a pilot scale

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ABSTRACT

Waste liquid mercury generated as a by-product of the Merrill–Crowe process in gold mining and recovered from mercury-containing waste must be stabilized for secure storage or disposal. This study developed a procedure for mercury stabilization. A ball mill with a 0.5 m³ capacity and a rotational speed of 43 rpm was used to stabilize the residual mercury with sulfur. The treatments were conducted for 30, 60, and 90 min at mercury: sulfur molar ratios of 1.0, 0.8, and 0.67. The ball loading ratio was 7.0 with residual mercury, and the temperature was below 40 °C. The treatment efficiency was evaluated by measuring the concentrations of mercury and other metals using the Toxicity Characteristic Leaching Procedure (TCLP), examining the stabilized residual mercury by X-ray diffraction, and conducting bioassays on *Daphnia magna* and *Lactuca sativa*. Principal component analysis (PCA) was performed on the aforementioned variables. The 90-min treatment, with a mercury-to-sulfur molar ratio of 0.67, stabilized mercury mainly as cinnabar compared with the other treatments and presented leachate mercury values below the detection limit <0.003. The leachate from the treatments also showed values of 21.28–38.44 toxic units, classified as very toxic, and generated toxicity, particularly for *D. magna*, because of the presence of other metals such as Al, Ba, B, Ca, Cu, Cr, Fe, Mn, Ni, and Zn. The variability of the residues in the PCA analysis was explained by the treatment effect and the presence of other metals in the residual mercury. The stabilized residual mercury obtained was classified as non-hazardous and could be stored or disposed of as ordinary waste in a security landfill.

1. Introduction

Residual mercury is a by-product of ore processing, such as gold, using the Merrill–Crowe technique. The ratio between gold and

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residual mercury in extraction can range from 1.0:1.0 to 2.93:1.0 [1,2]. Based on the official global gold production in 2017, an estimated residual mercury production of 2500 tons must be stabilized for disposal or storage [3].

The stabilization of residual mercury reduces its volatility and solubility by preventing it from entering the atmosphere, soil, and water through leaching [4,5]. Therefore, stabilization treatments form a species with more stable physical properties, such as mercury sulfide. Several methods are used for mercury stabilization. One uses sulfur to form mercury sulfide, such as metacinnabar and cinnabar. Another method is stabilization, which is performed by forming an amalgam with nonferrous metals [4,6,7] or encapsulation in an insoluble matrix, such as Portland cement, Sorel cement, silicates, and calcium aluminates. It can also be stabilized as mercury selenide [6,8]. Stabilization in countries such as the USA uses thermal roasting treatment. In the European Union, mercury must be converted to HgS, and the excess can be disposed of as Hg in salt mines. In Japan, mercury must undergo a stabilization/solidification process and be disposed of in isolated landfills [3].

A stabilization treatment using mercury sulfide generates a stable product with a high melting temperature, ranging from 386 °C to 583 °C. In addition, the compound has a high vapor pressure and low solubility in water [9]. Mercury sulfide exists as red cinnabar (α -HgS) and black metacinnabar (β -HgS). Red cinnabar is more stable, whereas metacinnabar is metastable (intermediate compound) [6,10]. In stabilization, several authors have used stoichiometric and non-stoichiometric molar ratios for mercury and sulfur ranging from 1.0:1.0 to 1.0:6.25 [11,12].

The present investigation used a 0.5 m³ ball mill with 0.05 m diameter balls and a rotational speed of 43 rpm; which is in contrast to other authors who worked with 0.00025 m³ mills, 0.02 m balls, and rotational speed between 200 and 400 rpm [11,13]. The higher mass of the balls and the lower rotational speed favor the cascading of the balls that generate sufficient energy for the reaction between residual mercury and sulfur, avoiding unnecessary energy increases and favoring the formation of cinnabar. By working with a mass of 15 kg of residual mercury per batch, the results are close to an industrial process.

The ball mill is suitable for stabilizing residual mercury, such as mercury sulfide. The movement of particles inside the mill and the contact zones of the colliding balls release large amounts of energy that can be harnessed for the development of the HgS reaction, which requires a moderate activation energy, which can vary between 27.2 and 184.4 kJ/mol depending on the type of sulfur used [14,

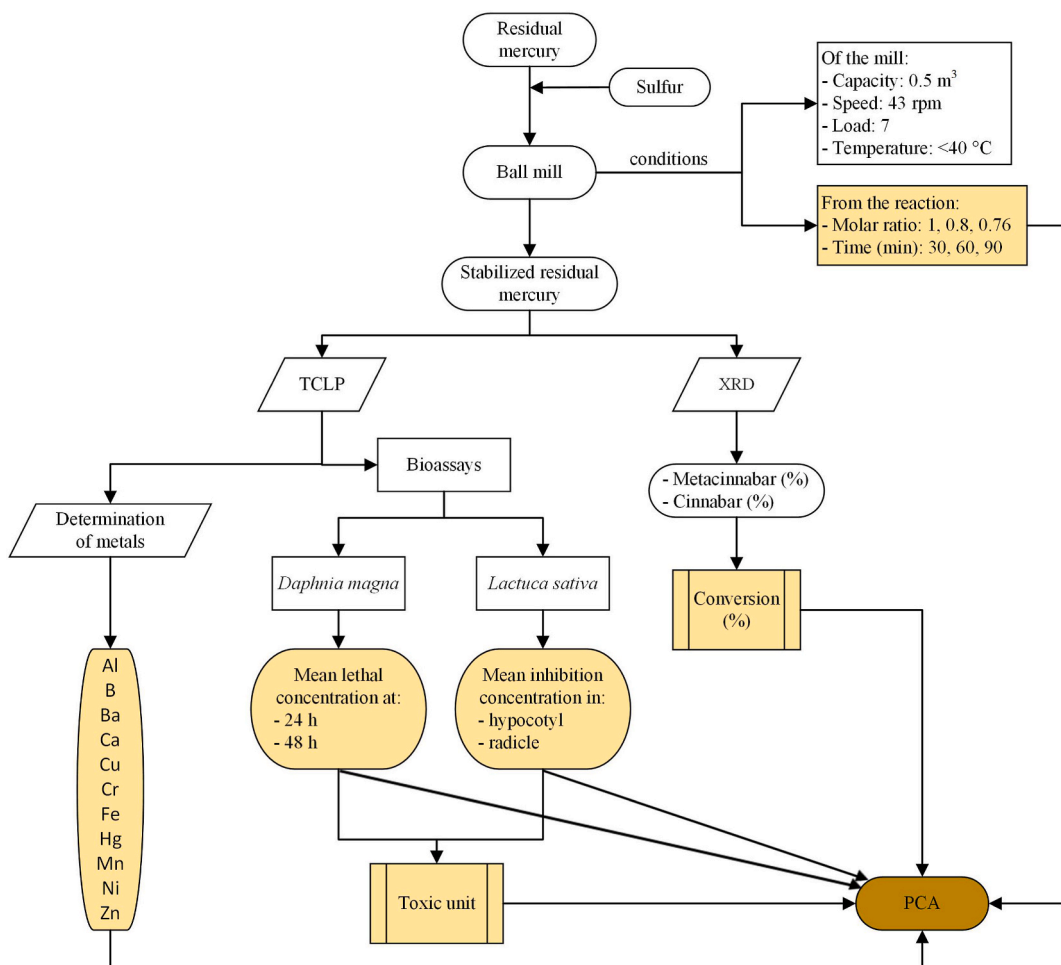


Fig. 1. Residual mercury stabilization methodology.

15]. Unlike other studies, the present study used stabilization tests at the pilot level using a ball mill at low rotation speeds, larger balls, and a greater mass of mercury treated per batch (15 kg), in addition to evaluating the use of residual mercury and the ecotoxicological effects of the stabilized material.

Studies on the ecotoxicity of mercury have determined a substantial effect at the level of *Lactuca sativa* radicles, expressed as the growth inhibitory concentration (IC₅₀), due to the formation of phytochelatin that prevent the entry of nutrients [16–18]. However, its toxicity on other organisms, such as *Daphnia magna*, is not as pronounced as that of Al, Zn, and Cr among other metals that precipitate at the gill level by a change in pH [17,19,20] or as boron, which has a toxic effect by riboflavin complexation [21]. The integral evaluation of the toxicity of a substance on different organisms is evaluated using toxic units, a dimensionless variable consisting of the inverse of the ecotoxicological parameters, and it can be averaged for all the effects evaluated. The scale of toxic units classifies wastes as non-toxic <1, toxic 1–10, very toxic 11–100, and extremely toxic >100 [22,23].

Mercury stabilization is considered adequate when the mercury concentration in the Toxicity Characteristic Leaching Procedure (TCLP) test leachate is less than 0.2 mg/L. This result suggests that the product is non-toxic and can be disposed of as ordinary waste in a security landfill [24] or when its effects on living organisms are in the non-toxic range evaluated as toxic units [17,22].

In this investigation, the stabilization of mercury as metacinnabar and cinnabar was evaluated under different reaction times and molar ratios of mercury to sulfur. The concentrations of mercury and other metals in the leachate from the TCLP test were examined by X-ray diffraction (XRD), and an ecotoxicological analysis of the leachate from the stabilized residual mercury was performed.

2. Materials and methods

2.1. Sample collection

Residual mercury samples were collected from the Tower and Tower SA Huatiquemer security landfill warehouse in Chincha, Peru, between May and September 2021. The origin of these samples is diverse, such as recovering residual mercury through evaporation.

2.2. Stabilization of residual mercury as a mercury sulfide

Stabilization of the residual mercury samples with sulfur was performed using a ball mill with a 0.5 m³ capacity, 5 cm diameter balls, a ball-to-residual mercury weight ratio of 7.0 [12], a rotation speed of 43 rpm [14], and Hg:S molar ratios of 1.0 (1.0:1.0), 0.8 (1.0:1.25), and 0.67 (1.0:1.50) at 23 °C and rotation times of 30, 60, and 90 min. The stabilized residual mercury was characterized by determining metals in the leachate to evaluate its toxicity and by performing mineralogical analysis to evaluate the percentage of cinnabar and metacinnabar (Fig. 1).

2.3. Characterization of stabilized residual mercury

The stabilized residual mercury leachates were obtained using the EPA method 1311, TCLP, and the determination of metals using the EPA method 6020B (Thermo Scientific ICP-optical, 6500 Duo, Germany).

The mineralogical analysis was performed by XRD (D2 PHASER, Bruker, Germany). Samples smaller than 63 μm were examined using an incident K-Alpha wavelength and a copper (Cu) anode. The data were taken over a scanning range (2θ) of 5.01°–79.99°, with a step size of 0.02°, using the ASTM C1721-15 method.

2.4. Ecotoxicity of the stabilized residual mercury

Ecotoxicity testing of the TCLP leachate was conducted according to the protocol for short-term toxicity testing of hazardous waste landfills (EPA 600/3–88/029). The median lethal concentration LC₅₀ at 24 and 48 h for *Daphnia magna* and the mean inhibition concentration IC₅₀ of hypocotyl and radicle in *Lactuca sativa* were determined. The LC₅₀ and IC₅₀ values were used to determine the toxic units (TU) of stabilized residual mercury [25]. TU is defined as the inverse of the LC₅₀ or IC₅₀ value expressed as the percentage dilution of the leachate multiplied by 100. The results were classified using the scale proposed by Persoone et al. [22], less than 0.4, non-toxic (nt); between 0.4 and 1.0, slightly toxic (lt); between 1 and 10 toxic (t); between 10 and 100, very toxic (mt); and greater than 100 TU, extremely toxic (et).

2.5. Statistical analysis

All tests were conducted in triplicate. The mean value and standard deviation are reported. The normal distribution of the values was tested using the Shapiro–Wilk test. The Kruskal–Wallis test and the multiple pairwise comparison test using Dunn's procedure were used to compare the individual means. Principal component analysis (PCA) was used to evaluate the correlations between the variable times, molar ratio, concentration of metals in the leachate (Al, Ba, B, Ca, Cu, Cr, Fe, Mn, Hg, Ni, and Zn), conversion percentage, LC₅₀ at 24 and 48 h for *Daphnia magna*, IC₅₀ for *Lactuca sativa* radicle, and toxic unit. XLSTAT software version 2023.2.1414 (Addinsoft, NY, USA) was used, with a significance level of 5 % in all cases.

Specialized Probit regression analysis, which transforms the concentration-response curve into a line that can be analyzed by least squares or maximum likelihood [26], was used to determine the LC₅₀ in *D. magna* at 24 and 48 h and the IC₅₀ of the radicle and hypocotyl of *L. sativa*.

3. Results and discussion

3.1. Stabilization of the residual mercury

The results of Hg concentrations after stabilization treatments at 30, 60, and 90 min and the molar ratios of 1.00, 0.80, and 0.67, are presented in Table 1. T1, T2, and T3 treatments at 30 min were unsuitable because numerous non-stabilized metallic mercury particles were observed. The 60-min stabilization process with a molar ratio of 1.00 (T4) showed an average mercury value of 2.184 mg/L in the TCLP leachate, which is higher than the USEPA 40 CFR Part 261 standard (0.2 mg/L). At 60-min, with molar ratios of 0.80 (T5) and 0.67 (T6), mercury concentrations were lower than 0.003 mg/L in the TCLP leachate. These values were similar to those reported by Lopez et al. [12] (0.0031 mg/L) at the same treatment time, indicating that the process is favored by excess sulfur. Furthermore, these values were higher than those reported by Fukuda et al. [11] (0.0003 mg/L), who used a laboratory planetary mill with a 250 cm³ capacity, with higher rotation speed (400 rpm), temperature, and time conditions similar to those of the developed process. Therefore, a larger mill size would require a longer time to complete the reaction.

The 90-min stabilization processes (T7, T8, and T9 for molar ratios 1.00, 0.80, and 0.67, respectively) showed mercury concentrations lower than 0.003 mg/L in the TCLP leachate, similar to those obtained by Lopez et al. [12].

In addition, detectable concentrations of Al, Ba, B, Ca, Cu, Cr, Fe, Mn, Ni, and Zn were observed in the TCLP leachate from the stabilized residual mercury (Table 2). Treatment T4 presented higher Mn, Ni, and Zn concentrations than the other treatments. T7 showed a higher Al concentration and higher B, Ca, Cr, and Fe concentration than T9. The presence of these metals was attributed to the residual origin of the mercury used. Zn comes from the Merrill–Crowe process, where Zn powder is used to precipitate valuable metals and mercury [27]. The presence of metals complies with the USEPA 40 CFR Part 261 regulations. However, it influences the ecotoxicological tests, affecting the development of the analyzed individuals [17,28].

3.2. HgS formation and conversion to metacinnabar and cinnabar

The T4, T5, T6, T7, T8, and T9 treatments stabilized the residual mercury as metacinnabar and cinnabar (Table 3). Fig. 2 shows the XRD patterns of treatments T4 and T9. In T9 (Fig. 2b), the concentration of metacinnabar no longer exists, as evidenced by the light color of the sample, unlike the dark color of sample T4 (Fig. 2a), which had a higher metacinnabar content than cinnabar. In addition, other minerals such as hawleyite (CdS), were detected in the treatments because of the use of zinc dust in the Merrill–Crowe processes, which favors the cementation of Cd and other elements [29]. T8 and T9 treatments, using a molar ratio of 0.8 and 0.67, respectively, and longer time (90 min), induced higher conversion to cinnabar [11,12]. The higher presence of cinnabar indicates better treatment because it is more stable than metacinnabar [10]. Similar to Dickson [10], metacinnabar is converted to cinnabar in the presence of sulfur for long periods. Similarly, the conversion of metacinnabar to cinnabar is favored when there is an excess of sulfur because cinnabar has a higher sulfur ratio in its octahedral crystal structure (HgS₆) than metacinnabar with a tetrahedral structure (HgS₄) [30]. Conversely, Fukuda et al. [11] worked with a higher rate and lower volume than this study. They obtained amorphous mercury sulfide species whose structure is in the process of formation to obtain metacinnabar.

3.3. Ecotoxicity results and toxicological units

The results of the ecotoxicological tests and toxicological units for *Daphnia magna* and *Lactuca sativa* are shown in Table 4. The T4 treatment had the greatest effect on *Daphnia* at 24 h (LC₅₀ = 1.64 %) and a greater effect at 48 h (LC₅₀ = 1.29 %), which is due to the combined effect of higher concentrations of Hg and other metals such as Mn, Ni, and Zn, from the leachate that upon entering the gills of the *Daphnia* sediments and causes a toxic effect on the individual [17,19]. Conversely, when evaluating the effect on *L. sativa*, in the T4 treatment, radicle growth was inhibited (IC₅₀ = 29.13 %) because of the presence of Hg (2.184 mg/L), which induces the formation of phytochelatin at the radicle level as a defense mechanism of the plant [16,17]. In general, *L. sativa* is more sensitive to inhibiting root growth against contaminants than other plant species [31–33]. In treatments T5, T6, T7, T8, and T9, there was no toxic effect on *L. sativa* because of the low Hg concentration in the leachate. Other authors only found high toxic effects when the Hg concentration

Table 1

Mercury concentration in TCLP after stabilization treatments. Mercury data in TCLP are expressed as mean ± standard deviation (n = 3).

Treatment	Time (minutes)	Molar ratio	Mercury in TCLP (mg/L)
T1	30	1.00	Non-stabilized
T2	30	0.80	Non-stabilized
T3	30	0.67	Non-stabilized
T4	60	1.00	2.184 ± 0.8 ^b
T5	60	0.80	<0.003 ^a
T6	60	0.67	<0.003 ^a
T7	90	1.00	<0.003 ^a
T8	90	0.80	<0.003 ^a
T9	90	0.67	<0.003 ^a

^{a, b} letters in the mercury content by the TCLP column imply a statistical difference (p < 0.05) according to multiple pairwise comparisons using Dunn's procedure.

Table 2Metal content of the TCLP leachate. Data are expressed as mean \pm standard deviation (n = 3).

Treatment	Al	Ba	B	Ca	Cu	Cr	Fe	Mn	Ni	Zn
	mg/L									
T4	0.071 \pm 0.022	0.015 \pm 0.005	0.092 \pm 0.013	1.700 \pm 0.728	0.009 \pm 0.005	0.010 \pm 0.010	0.253 \pm 0.098	0.059 \pm 0.021	0.149 \pm 0.126	0.252 \pm 0.021
T5	0.011 \pm 0.006	<0.005	0.272 \pm 0.254	0.282 \pm 0.151	<0.006	0.008 \pm 0.002	0.640 \pm 0.280	0.034 \pm 0.016	0.009 \pm 0.004	<0.006
T6	0.008 \pm 0.005	<0.005	0.589 \pm 0.265	0.719 \pm 0.118	<0.006	0.008 \pm 0.001	1.177 \pm 1.026	0.017 \pm 0.006	<0.005	<0.006
T7	0.170 \pm 0.156	0.012 \pm 0.007	1.100 \pm 0.483	4.099 \pm 3.193	<0.006	0.009 \pm 0.005	0.140 \pm 0.080	0.043 \pm 0.040	0.008 \pm 0.003	0.007 \pm 0.001
T8	0.036 \pm 0.015	<0.005	0.530 \pm 0.155	0.237 \pm 0.054	<0.006	0.004 \pm 0.001	0.060 \pm 0.001	0.017 \pm 0.001	<0.005	<0.006
T9	0.031 \pm 0.026	<0.005	2.109 \pm 1.290	8.119 \pm 6.588	<0.006	0.018 \pm 0.002	1.810 \pm 0.900	0.032 \pm 0.007	0.008 \pm 0.003	0.011 \pm 0.005

Table 3Results of the X-ray diffraction analysis. The data are expressed as mean \pm standard deviation (n = 3).

Treatment	Time (minutes)	Molar ratios	Metacinnabar β -HgS (%)	Cinnabar α -HgS (%)	Others (%)	Conversion of metacinnabar to cinnabar (%)
T4	60	1.00	60.85 \pm 6.55	20.65 \pm 4.85	18.5 \pm 1.70	25.43 ^a
T5	60	0.80	47.54 \pm 4.19	38.30 \pm 1.84	14.17 \pm 2.35	44.67 ^{a,b}
T6	60	0.67	14.22 \pm 1.45	26.49 \pm 1.36	59.30 \pm 2.81	65.11 ^{a,b}
T7	90	1.00	32.55 \pm 1.62	19.56 \pm 0.83	47.89 \pm 2.45	37.54 ^{a,b}
T8	90	0.80	9.42 \pm 1.37	25.97 \pm 3.06	64.62 \pm 4.43	73.41 ^{a,b}
T9	90	0.67	0.0 \pm 0.0	50.58 \pm 3.28	49.42 \pm 3.28	100.00 ^b

^{a, b} letters in the converting the metacinnabar to cinnabar column imply a statistical difference ($p < 0.05$) according to multiple pairwise comparisons using Dunn's procedure.

was between 0.364 and 4.148 mg/L [31,34].

The toxic units (TU) of the ecotoxicological tests for *D. magna* and *L. sativa* as evaluation organisms presented values between 21.28 and 38.44, which, according to the scale of Persoone et al. [22], were classified as very toxic. The order of toxicity of the treatments was T8 < T5 < T6 < T7 < T9 < T4. T4 treatment presented a higher TU (38.44) because of the shorter stabilization time (60 min), higher molar ratio of Hg:S (1.0), and higher concentrations of Hg, Ni, and Zn in the TCLP leachate. The treatment with the best result was T8 because of a longer stabilization time (90 min), lower Hg:S molar ratio (0.8), absence of Hg, and low Cr, Fe, Mn, Ni, and Zn concentrations in the TCLP leachate.

3.4. Principal component analysis

PCA was performed for the variables time, molar ratio, concentration of metals in the leachate (Al, Ba, B, Ca, Cu, Cr, Cr, Fe, Mn, Hg, Ni, and Zn), conversion percentage, LC₅₀ at 24 and 48 h for *D. magna*, IC₅₀ for *L. sativa* radicle, and toxic unit (Table 5). Eighteen variables were considered, mostly distributed in factors F1 (51.25 %) and F2 (79.87 %) (Fig. 3).

Factor F1 correlated positively with the molar ratio of Ba, Cu, Mn, Hg, Ni, and Zn in the TCLP leachate, with toxic unit value on *D. magna* and *L. sativa*, and negatively with the percentage conversion of metacinnabar to cinnabar, with the LC₅₀ at 24 and 48 h for *D. magna*, and with the IC₅₀ of the radicle for *L. sativa*. The treatment effect was observed in this factor, indicating that a lower molar ratio results in a higher conversion of residual Hg to cinnabar, i.e., stabilized Hg is obtained. In contrast, a higher amount of non-stabilized residual mercury indicated a greater presence of metals, such as Ba, Cu, Mn, Ni, and Zn, in the leachate, which generates toxicity to *D. magna*, and the presence of Hg in the leachate generates toxicity in the radicle of *L. sativa*. Toxicity in these individuals increases the value of the toxic unit.

Factor F2 positively correlates the content of metals, such as B, Ca, Cr, and Fe in the TCLP leachate with the value of the toxic unit on *D. magna* and *L. sativa* and negatively correlates with the LC₅₀ at 24 and 48 h for *D. magna*. This factor shows the effects of the leached metals present in the residual mercury samples and their influence on the ecotoxicity of *D. magna*. Boron has a toxic effect on *D. magna* due to riboflavin complexation, which interferes with its metabolism [21]. Conversely, Cr and Fe, when entering the gills of these individuals, precipitate as hydroxides caused by pH variations, generating a toxic effect [20].

PCA revealed the concentration of metals in the leachate, conversion percentage, and ecotoxicological tests of the stabilization treatments.

4. Conclusions

Stabilization of the residual mercury as mercury sulfide using a 0.5 m³ ball mill with a rotational speed of 43.5 rpm was achieved

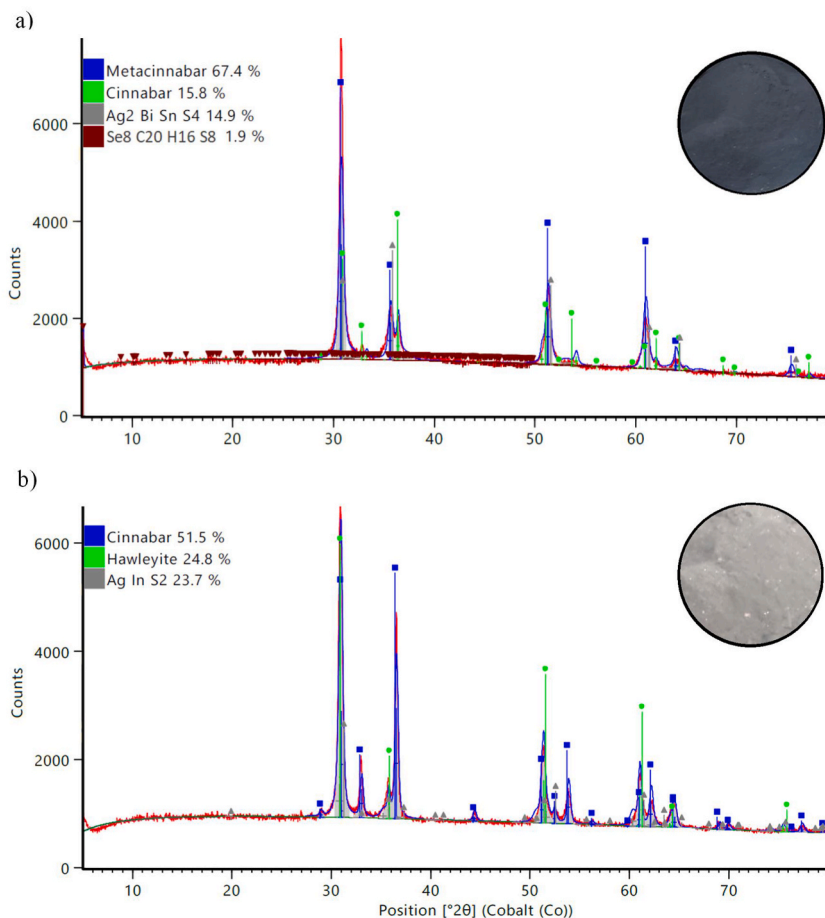


Fig. 2. XRD patterns of the stabilized residual mercury from a) treatment T4 and b) treatment T9.

Table 4

Results of ecotoxicological tests and toxic units for *Daphnia magna* and *Lactuca sativa*. The data are expressed as mean \pm standard deviation ($n = 3$).

Treatment	Ecotoxicological tests				Toxic units (TU)				TU
	<i>Daphnia magna</i>		<i>Lactuca sativa</i>		<i>Daphnia magna</i>		<i>Lactuca sativa</i>		
	% LC ₅₀ (24 h)	% LC ₅₀ (48 h)	% IC ₅₀ hypocotyl	% IC ₅₀ radicle	LC ₅₀ (24 h)	LC ₅₀ (48 h)	IC ₅₀ hypocotyl	IC ₅₀ radicle	
T4	1.64 \pm 0.40 ^a	1.29 \pm 0.42 ^a	100.00 \pm 0.00	29.13 \pm 10.29 ^a	63.75	85.23	1.00	3.80	38.44
T5	2.58 \pm 0.43 ^a	2.09 \pm 0.06 ^a	100.00 \pm 0.00	100.00 \pm 0.00 ^b	39.44	47.92	1.00	1.00	22.34
T6	2.83 \pm 0.41 ^a	1.84 \pm 0.58 ^a	100.00 \pm 0.00	100.00 \pm 0.00 ^b	35.8	58.22	1.00	1.00	24.00
T7	2.89 \pm 0.13 ^a	1.52 \pm 0.03 ^a	100.00 \pm 0.00	100.00 \pm 0.00 ^b	34.66	66.03	1.00	1.00	25.67
T8	2.76 \pm 0.16 ^a	2.13 \pm 0.03 ^a	100.00 \pm 0.00	100.00 \pm 0.00 ^b	36.24	46.88	1.00	1.00	21.28
T9	1.79 \pm 0.63 ^a	1.33 \pm 0.24 ^a	100.00 \pm 0.00	100.00 \pm 0.00 ^b	61.10	75.75	1.00	1.00	34.96

LC₅₀ = median lethal concentration, IC₅₀ = median inhibitory concentration. ^{a, b} letters in the same LC₅₀ and IC₅₀ column imply statistical difference ($p < 0.05$) according to multiple pairwise comparisons using Dunn's procedure.

using a treatment time of 90 min with a mercury-to-sulfur molar ratio of 0.67. The result was a mercury content in the TCLP leachate below the detection limit (<0.003 mg/mL), consisting mainly of cinnabar and metacinnabar. Therefore, it is classified as non-hazardous waste because it is below 0.2 mg/mL, which can be stored or disposed of as ordinary waste in a security landfill. The presence of B, Ba, Ca, Cu, Cr, Fe, Mn, Ni, and Zn is responsible for the adverse effects in the bioassays, particularly in *D. magna*, whereas Hg is responsible for the toxic effects in *L. sativa*.

Data availability statement

Data will be made available on request.

Table 5

Principal component analysis of the concentration of metals in the leachate, conversion percentage, and ecotoxicological tests of the stabilization treatments.

	F1	F2
Time (min)	-0.363	0.375
Molar ratios	0.734	-0.341
Al-leached TCLP	0.355	-0.024
Ba-leached TCLP	0.911	-0.064
B-leached TCLP	-0.394	0.863
Ca-leached TCLP	-0.035	0.940
Cu-leached TCLP	0.966	-0.017
Cr-leached TCLP	0.104	0.966
Fe-leached TCLP	-0.367	0.771
Mn-leached TCLP	0.901	0.152
Hg-leached TCLP	0.966	-0.017
Ni-leached TCLP	0.970	-0.007
Zn-leached TCLP	0.966	0.000
Conversion of metacinnabar to cinnabar (%)	-0.685	0.599
<i>Dafnia magna</i> LC ₅₀ (24 h)	-0.638	-0.640
<i>Dafnia magna</i> LC ₅₀ (48 h)	-0.628	-0.705
<i>Lactuca sativa</i> IC ₅₀ radicle (%)	-0.966	0.017
Toxic unit	0.733	0.667
Cumulative (%)	51.246	79.865

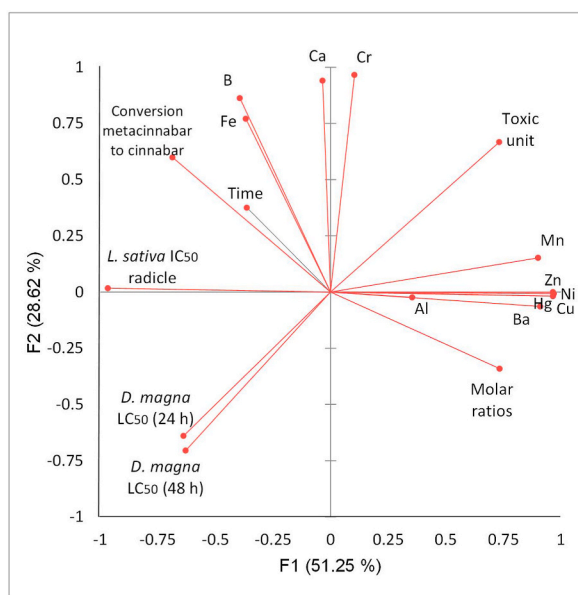


Fig. 3. PCA biplot of the variables time, molar ratio, concentration of metals in the leachate (Al, Ba, B, Ca, Cu, Cr, Cr, Fe, Mn, Hg, Ni, and Zn), conversion percentage, LC₅₀ at 24 and 48 h for *D. magna*, IC₅₀ for *L. sativa* radicle and toxic unit.

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CRedit authorship contribution statement

Pablo Cesar Neyra Avellaneda: Validation, Investigation, Formal analysis, Data curation. **Reider Benigno Zarate Sulca:** Validation, Investigation, Formal analysis, Data curation. **Hilda Beatriz Magallanes Camasca:** Supervision, Resources, Methodology, Investigation. **Bernardette Mariella Sencebe Bastante de Garcia:** Resources, Project administration. **Javier Fortunato Pujaico Lizarbe:** Resources, Methodology, Investigation. **Lena Asunción Téllez Monzón:** Methodology, Investigation. **Lisveth Flores del Pino:** Methodology, Investigation. **José Edmundo Huerta Alatrística:** Resources, Funding acquisition. **Paola Jorge-Montalvo:** Writing – review & editing, Writing – original draft, Validation, Formal analysis. **Lizardo Visitación-Figueroa:** Writing – review &

editing, Writing – original draft, Supervision, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Pablo Neyra Avellaneda reports financial support was provided by National Innovation Program for Competitiveness and Productivity for Business Development Category 1, Innovate Peru of the Ministry of Production. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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