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Research Article

Solidification and stabilization of mercury-contaminated tailings in artisanal and small-scale gold mining using tras soil

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Abstract

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Artisanal and small-scale gold is known to be sources of mercury pollution. This mercury contamination occurs when gold is isolated by the amalgamation method, contamination occurs. Mercury pollution in smallscale artisanal gold mining in the Kulon Progo area of Yogyakarta, the lowest tailing content was 164.49 mg kg⁻¹, and the highest was 383.21 mg kg⁻¹. This value exceeded the quality standard stipulated by Indonesian Government Regulation 22 of 2021 of 75 mg kg⁻¹. The technology that can control pollution is stabilization/solidification (S/S). This research aimed to determine the optimum composition of the tailings mixture with tras soil. Variations in the design of tras soil with tailings were 100:0, 90:10, 80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 20:80, and 10:90. The results showed that the optimum tras: tailings soil composition was 90:10, with a compressive test of 31 t m⁻². Toxicity Characteristic Leaching Procedure (TCLP) was 0.0033 mg L⁻¹ according to the quality standard of Indonesian Government Regulation No. 22, 2021, with a value of 0.05 mg L⁻¹. The compressive strength results follow the quality of the US EPA of 35 t m^{-2} .

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Introduction

Activities in small-scale artisanal gold mining contribute to mercury (Hg) pollution (Esdaile and Chalker, 2018). The use of mercury began 30 years ago because it is increasingly difficult to separate gold and mining rocks (Tschakert and Singha, 2007). The use of mercury that exceeds the threshold can pose a grave danger. The Minamata case that occurred in the 1960s is one of the events that explain the dangers of mercury to living things (Ekino et al., 2007). Mercury pollution also hurts the environment and aquatic organisms because it is deadly to the biota that lives in these waters (Pandey and Madhuri, 2014).

According to Rachman et al. (2017), the Kulon Progo gold processing process usually uses amalgamation techniques. This amalgamation involves mixing mercury with water, soil, or rock

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containing gold to form an amalgam (Rachman et al., 2018). According to Bose-O'Reilly (2008), the amalgamation process will produce pure gold and waste products in the form of tailings. Because it is straightforward and does not require the latest technology, many miners rely on this amalgamation procedure with mercury. This contamination occurs when gold is isolated by the amalgamation method (Drace et al., 2012). Based on Regulation of the Government of the Republic of Indonesia Number 22 (2021).concerning the Implementation of Environmental Protection and Management, tailings from mineral ore processing are included in the list of hazardous and toxic waste materials from specific sources. Tailings has a waste code of B416 which falls into category 2. Tailings from the Kulon Progo gold mining amalgamation process are channelled into

a holding tank. However, it was also explained that the reservoir's capacity is insufficient to accommodate the existing tailings. This condition resulted in tailings spillage so that the tailings flowed into the yard (Rachman et al., 2018). Tailings must meet the storage requirements following the commandment of the Head of the Environmental Impact Management Agency Number 1 of 1995 concerning Procedures and Technical Requirements for the Storage and Collection of Toxic and Hazardous Waste.

Rachman et al. (2018) in their study on mercury pollution in small-scale artisanal gold mining in the Kulon Progo area of Yogyakarta, reported the most negligible tailing content is 164.49 mg kg⁻¹, and the largest is 383.21 mg kg⁻¹. According to the regulations, the permissible concentration of mercury in tailings is 75 mg kg⁻¹. This value exceeds the quality standard stipulated by the Government Regulation of the Republic of Indonesia No. 21 of 2021. Hg has high and volatility, as well as easy toxicity bioaccumulation. Methylated Hg has a high affinity for fatty tissues in organisms and can accumulate through the food chain to more toxic levels in these organisms (Singh and Kalamdhad, 2011). Methyl Hg has a solid binding capacity to proteins in the bodies of aquatic animals and plants, with the bioaccumulation and biomagnification of Hg can endanger human health (Kim et al., 2016). Based on the existing condition of tailings disposal that forms a heap, it is necessary to have a tailings pile processing technique to reduce pollution following the established quality standards.

One of the efforts that can be made to reduce pollution from mercury-contaminated product discharges is the solidification/stabilization (S/S) technology. According to Misra and Pandey (2005), working with S/S technology can detoxify the toxic content in waste and limit contamination from hazardous waste compounds. The advantages of hazardous and toxic waste treatment with S/S technology, in addition to the technology being easy to apply, environmentally friendly, and low operating costs (Huang et al., 2017). Hazardous and toxic waste using S/S technology is converted into a solid compound so that no leachate occurs. S/S technology requires an additional binder in the form of a pozzolan to encapsulate contaminants both chemically and chemically to become a more stable form (Chen et al., 2019).

Tras soil is one of the natural pozzolans used in the S/S technique because it has pozzolanic properties (Raj et al., 2005). Tras is a raw pozzolanic material mainly consisting of reactive silica or aluminate. Tras soil mixed with water and lime will form a solid, hard, and insoluble mass in water (Morsy et al., 2012). According to Karamalidis et al. (2008), tras soil has properties when mixed with extinguished lime and water, will have cement-like properties. As a type of pozzolan, tras soil has cement-like properties; tras soil has the potential as a pozzolan because it can isolate the mobility of heavy metals and provide a positive correlation as an additive (Shammas, 2017). Using tras soil as an additive in portland cement mixtures in the S/S application acts as a binder that can isolate the movement of heavy metal contaminants and provide increased mechanical strength (Correia, 2020)

This study aimed to obtain the best composition between tailings and tras soil mixture at artisanal and small-scale gold mining in Kulon Progo Yogyakarta with environmental quality standards based on applicable regulations.

Materials and Methods

Materials

Tras soil was imported from PT Semen Indonesia, Gresik. Samples were taken at the Kulon Progo Gold Mine Location. According to Palar et al. (2013), the chemical composition of tras soil consists of silica (SiO₂) of 50%, aluminum oxide (Al₂O₃) of 16%, crystal water (H₂O) of 10%, iron oxide (Fe₂O₃) of (8%), calcium oxide (CaO) of 3%, magnesium oxide (MgO) of 3%, sodium oxide (Na₂O) of 5% and calcium oxide (K₂O) of 5%. Control soil samples were taken from two locations not contaminated with mercury and then in the composite. Meanwhile, tailings samples were taken from 5 tailings ponds. Each piece in the control soil and tailings pond was taken at a depth of 90 cm, 60 cm, and 30 cm. Based on the soil depth, samples were taken at five tailings sites and control sites and then mixed based on the depth. Sampling was carried out using a sampling standard following US EPA rules. Each piece was placed in PET (Poly Ethylene Terephthalate) plastic and labeled, then placed in an ice box at 4 °C and brought to the laboratory for analysis of the total mercury concentration and tailings characteristics. The locations of the tailings sampling and control soil samples are shown in Figure 1.

Soil sample characterization

The particle test procedure based on ASTM D422 was as follows: (1) take a soil sample of 500 g, and check the water content, (2) place the filter assembly on top of the vibrating machine and insert it. The soil sample at the top of the arrangement was then closed tightly, (3) tighten the clamp and turn on the vibrating machine for 15 minutes approximately, and (4) weight each filter along with the retained soil sample.

pH value

The tailings pH determination was carried out using a pH meter. The tailings pH test procedure was as follows: (1) take enough tailings in the beaker glass, (2) add distilled water until 2/3 of the beaker glass is filled, (3) stir until homogeneous using a magnetic stirrer, and (4) measure the pH of the tailings using a pH meter.



Figure 1. The tailings sample location (Rachman et al., 2017).

Tailings characterization

Samples from the mine site were then analyzed for chemical properties in the form of pH testing, which was measured using a pH meter. The water content test was carried out by the gravimetric method. ASTM D422 is used to determine the physical characteristics of the soil based on the distribution of particles analyzed at the Soil and Rock Mechanics Laboratory of Civil Engineering FTSP ITS. Meanwhile, to determine the levels of mercury in the soil and tailings carried out at LPPT UGM Yogyakarta using Mercury Analyzer Type VM-3000.

Compressive strength test procedure

The compressive strength test procedure was based on ASTM C 109 concerning the standard test method for compressive strength of hydraulic cement mortars as follows: (1) remove the object to be tested from the mold, (2) turn on the machine for the compressive strength test, (3) set the loading capacity according to the object to be tested; note that the needle on the machine must be zeroed before it is ready for use,

(4) lift the loading hammer by turning the lifting knob, (5) place the specimen on the base of the loading hammer, (6) place the loading hammer until it touches the test object's surface, (7) rotate the loading hammer slowly until it presses against the test object. It is a loading speed of 1.4-3.4 kg cm⁻² sec⁻¹, (8) record the reading on the meter after the needle stops, and (9) tests on the specimens are carried out until the samples are destroyed.

Mercury analysis

The work procedure for the acid digestion method can vary according to the purpose or further analysis to be carried out. Nitric acid digestion according to Standard Method 3030-E concerning nitric acid digestion of metals samples was used for the digestion process of samples tested for metal content. Meanwhile, Standard Method 3050-B concerning acid digestion of sediments, sludges and soils for metals analysis by FLAA/ICP or GFAA/ICPMS was used if the results of digestion were to undergo FLAA/ICP or GFAA/ICPMS tests.

Nitric acid digestion according to Standard Method 3030-E

The procedure for the nitric acid digestion method according to US EPA Method 3050-E concerning acid digestion of sediments, sludges, and soils is as follows: (1) add liquid acid (perchloric, hydrochloric, or sulfuric acid) to the beaker containing the tailings sample until the volume reaches 100 mL, (2) add 5 mL of nitric acid (HNO₃) and close the beaker tightly, (3)boil the solution until the volume of the solution in homogeneous conditions reaches 10-20 mL, (4) continue heating until the digestion process is complete when the solution turns clear; to prevent the sample from drying out during the heating process, add sufficient HNO₃ to the solution, (5) filtrate the heated solution until a filtrate is obtained, (6) dilute the filtrate from the digestion result solution with distilled water until the volume becomes 100 mL, and (7) after chilling, take some filtrates to analyze the mercury level using the dithizone method.

Nitric acid digestion according to Standard Method 3050-B concerning acid digestion of sediments, sludges, and soils for metals analysis by FLAA/ICP or GFAA/ICPMS

The procedure for the nitric acid digestion method according to Standard Method 3050-B is as follows: (1) weigh 1-2 g (wet weight) or 1 g (dry weight) of the sample, (2) heat until a clear solution is formed; add a sufficient amount of nitric acid and hydrogen peroxide during heating to prevent the sample from drying out, (3) add 10 mL hydrochloric acid (HNO₃) and close the beaker tightly, (4) heat the solution to 95 °C \pm 5 °C and reflux for 10-15 minutes without heating, (5) add 5 mL of HNO₃ continuously until no brown smoke is formed, indicating the presence of the HNO₃ oxidation process in the sample, (6) evaporate the solution without heating until 5 mL remains or heat at 95 °C \pm 5 °C for 2 hours, (7) cool the sample and add 2 mL of distilled water and 3 mL of 30% H₂O₂, then cover, (8) reheat the solution and add 1 mL of 30% H₂O₂ until the solution is stable, (9) continue heating until 5 mL remains or heat at 95 °C \pm 5 °C for 2 hours, (10) add 10 mL HCl and cover, then reflux the solution at 95 $^{\circ}\mathrm{C}$ \pm 5 °C for 15 minutes, (11) filter the solution using Whatman paper No. 41 and put the filtrate into a 100 mL volumetric flask, and (12) add distilled water to the required volume for the AAS, ICP test or Mercury Analyzer.

Manufacture of test objects

The manufacture of test objects has a cube-shaped mold of 5 cm on each side. The test object was made with variations in the composition of tras and tailings soil, from the composition of 100:0; 90:10; 80:20; 30:70; 60:40; 50:50; 40:60; 30:70; 20:80, to 10:90. Mortar testing was carried out twice, then the average result was taken from the test. The process of treating test specimens from stirring, making specimens,

compacting and treating refers to SNI 2493:2011. The composition of the variation of tras and tailings soils can be seen in Table 1.

Composition (%)	Tras Soil (g)	Tailing (g)
100:0	300	0
90:10	270	30
80:20	240	60
70:30	210	90
60:40	180	120
50:50	150	150
40:60	120	180
30:70	90	210
20:80	60	240
10:90	30	270

Table 1. Binder composition variation.

Results and Discussion

Soil sample characteristics

The control soil and tailings at the artisanal and smallscale gold mining site in Kulon Progo are generally brown and yellowish and contain small amounts of gravel, clay, sand, and silt. For the sampling points, the coordinates of the five sampling points are presented in Figure 1. Chemical characterization and physical characteristics of the control soil and tailings are presented Tables 2 and 3.

Table 2. Grain distribution analysis on control soil.

Soil Fraction	Size (mm)	Percent (%)	Water (%)
Sand			
 Coarse 	≥2.00		
– Medium	≥0.425	39.36	
– Fine	≥0.075		19.16
Clay	≥ 0.0001	24.61	
Silt	≥0.0055	34.46	
Gravel	≥4.76	1.57	

Table 3. Grain distribution analysis on tailing	ngs.
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Soil fraction	Size (mm)	Percent (%)	Water (%)
Sand			
 Coarse 	≥ 2.00	55 17	
– Medium	≥0.425	33.17	
– fine	≥ 0.075		13.24
Clay	≥ 0.0001	12.27	
Silt	≥0.0055	30.69	
Gravel	≥4.76	1.86	

Based on Tables 2 and 3, the water content of the control soil is higher than that of the tailings. This is because the control soil contains less sand than the tailings, while the control soil contains more clay than the tailings, so water is retained in the clay. Clay soils

are known to have very slow permeability (Neuzil, 2019). Pore size is very decisive in soil permeability; the smaller the pores in the ground, the slower the soil permeability (Cao et al., 2021). Soil density significantly affects the entry of water into the soil. The results of mercury inspection in soil and tailings can be seen in Table 4. Based on Table 4, the pH of the

control soil was 6.74. on the other hand, the pH in the tailings samples varied from a low of 7.48 to a high of 8.56. The pH value not only affects certain metals in the soil but also affects changes in soil characteristics (Cheng et al., 2020). A high pH value will result in low metal solubility due to similar ions affecting the polluted soil (Zhang et al., 2020).

Sample code	Latitude/ longitude	рН	Total mercury concentration in tailing (mg kg ⁻¹)	Hg Quality Standards in TK-B* (mg kg ⁻¹)
CS-1	110°3'36.704" E 7°49'36.672" S	6.74	0.079	
TS-1	110°3'44.474" E 7°50'1.820" S	8.56	352. 32	
TS-2	110°3'45.615" E 7°50'1.628" S	7.76	326.66	75
TS-3	110° 3'56.786" E 7°50'16.648" S	7.88	164.49	75
TS-4	110° 3'57.644" E 7°50'15.453" S	8.18	251.51	
TS-5	110° 3'49.860" E 7°50'31.336" S	7.48	383.21	

Table 4. Mercury concentration and pH in soil and tailings.

*PP (Government Regulation) No. 22 of 2021 concerning the Implementation of Environmental Protection and Management.

According to Nortjé and Laker (2021), several environmental factors affect heavy metals: soil availability, organic matter, temperature, texture, clay minerals, elemental content, and others. Soil pH directly affects absorption, precipitation, complex forms, and oxidation-reduction reactions (Penn and Camberato, 2019). The mercury in the control soil sample is tiny compared to the mercury in the tailings sample. The mercury value in the control soil was 0.079 mg kg⁻¹. While the lowest mercury value in tailings is 164.49 mg kg⁻¹, and the highest is 383.21 mg kg⁻¹, the mercury value has exceeded the 75 mg kg⁻¹ standard set by Indonesian government regulation. According to Trihadiningrum et al. (2019), the result of the speciation of mercury at the artisanal and smallscale gold mining in Kulon Progo is 75% consists of volatile mercury (Hg⁰), with the occurrence of a hydrological cycle and the influence of wind direction; this causes the location of the control soil samples to contain mercury even in tiny amounts.

The spread of mercury in the control soil is caused by the hydrological cycle and is also influenced by wind speed and direction. The hydrological cycle plays a role in evaporation, condensation, and infiltration, supported by wind speed and direction (Ossai et al., 2020). The difference in the concentration of Hg in the tailings stockpile is not only caused by Hg spills from the gold processing process but is also influenced by soil characteristics, evaporation, condensation from the local area, and is influenced by surface runoff and runoff (Agboola et al., 2020). The difference in the concentration of Hg is also due to the characteristics of the soil (Liu et al., 2021). According to Laker and Nortje (2020), generally, the soil layer consists of sand, silt, and clay. Soils with sand and silt categories generally will be easier to pass water, whereas water flow in the ground will flow by infiltration or percolation through cracks and pores of soil and rock. At the same time, the clay type has impermeable properties where the surface layer sucks water until it is saturated, causing the mercury concentration to be trapped and trapped at that depth. Therefore, there are differences in the level of total mercury concentration at each sample location.

Compressive strength of tras soil

Based on the data in Table 5, it can be seen that the highest compressive strength value is found in the test object with a composition of 10% tras soil and 90% tailings at 118 t m⁻². Meanwhile, the lowest compressive strength value is at 90:10 composition, which is 31 t m⁻². The compressive strength test of all pieces meets the US EPA stand quality standards of 35 t m⁻². The compressive strength of the test object shows that the less addition of tras soil, the higher the compressive strength results. According to Kolawole et al. (2021), the use of pozzolan, in this case, is tras when used as a substitute for Portland cement which ranges from 10-35% of the weight of cement. The lower the percentage of tras soil, the less porosity. In 100% tras soil composition, cracks occurred during the 28-day curing period, which caused the compressive strength value to be low. Tras soil takes longer to dry and harden cement (Rachman et al., 2021). According to Santhosh et al. (2022), tras have properties that, when mixed with extinguished lime (the lime) and

water, will have cement-like properties. This property is caused by the amorphous silica oxide (SiO_2) and aluminum oxide (Al_2O_3) in the tras, making it acidic. Overall, concrete with a fine aggregate mixture performed better than standard concrete (Hafez et al., 2020). The composition of the tailings sample, which is more than the sample tras soil, will require excess water (Qi and Fourie, 2019).

Composition	Compressive Strength Value (t m ⁻²)			Quality standards
Tras Soil: Tailing (%)	Compressive Strength	Compressive Strength	Average Velue	$\frac{1}{1}$ compressive strongth* (t m ⁻²)
1 annig (70)	Objects 1	Objects 2	value	strength (t m)
100:0	27	22	25	
90:10	31	31	31	
80:20	71	85	78	
70:30	84	86	85	
60:40	76	98	87	
50:50	101	87	94	35
40:60	105	95	100	
30:70	87	115	101	
20:80	119	105	112	
10:90	111	124	118	
0:100	51	55	53	

Table 5. Compressive strength of tras soil and tailings

*US EPA Standard.

The tras soil will need excess water because the soil sample contains silt and clay, which has water-holding properties. Clay-structured soils have a large specific surface area. They have high water binding ability (Ignatavičius et al., 2022).

Toxicity Characteristic Leaching Procedure (TCLP) *test*

Based on the data in Table 6, the value of the TCLP Hg test results in all samples is far below the quality standard based on PP No. 22 of 2021, which is 0.05 mg L⁻¹. The TCLP values of some examples vary in some compositions. However, all models were below the limit of detection (LOD) <0.0005 mg L⁻¹, which means it was not detected (no detection) except for 60% tras composition and 40% tailings 0.0091 mg L⁻¹. The values listed below are the LOD TCLP test results because the detection tool from the test is still in the noise position. In addition, the inclusion of numbers under the LOD is used for research purposes. The low mercury TCLP result value could occur due to the influence of pH in the TCLP test.

The low value of mercury TCLP results can occur because of the influence of pH on the TCLP test. The initial pH value of the tailings is in the range of 6.74-8.56. The results of the calculation of the coefficient (Q) Hg(OH)₂ to the value of Ksp Hg(OH)₂, the value of Q>Ksp. If the coefficient value is more significant than Ksp, then Hg(OH)₂ residue occurs, so the tool does not read Hg²⁺. Because the device cannot read the change in mercury type, the TCLP value in the tailings is minimal and undetectable. Increasing tras soil as pozzolan in the test object can reduce the TCLP value. A low TCLP test result means that the concentration of mercury leachate in the test object will also be low.

Table 6.	TCLP and Hg concentration of trans soil and
	tailing.

Composition Tras Soil: Tailing (%)	Hg Concentration (mg L ⁻¹)	TCLP Quality Standard * (mg L ⁻¹)
100:0	0.0017	
90:10	0.0033	
80:20	0.0006	
70:30	0.0005	
60:40	0.0091	0.005
50:50	0.0018	0.005
40:60	0.0014	
30:70	0.0001	
20:80	ND	
10:90	0.0009	

Notes: *PP No. 22 of 2021, ND = not detected.

The mercury concentration is low because the mercury is bound in the pozzolan, in this case, the tras soil. In tras soil, there is a reaction between carbonate and essential compounds, which results in mercury being chemically bound (Yousuf et al., 1992). Elemental mercury crystals are formed when mercury reacts with an alkaline solution. Even if an alkaline solution is added, mercury in the elemental form will not dissolve (Donatello et al., 2012). The hydration process produces Ca(OH)₂, a potent base compound. Ca(OH)₂ reacts with Hg²⁺ and forms Hg⁰ crystals according to the above equation. Hg⁰ crystals are more stable than Hg²⁺. In the process of calcium silicate hydrate matrix and calcium aluminate hydrate, Hg⁰ binding occurs. The mercury in the hydroxide precipitate and the carbonate salts are bound in an insoluble crystalline structure. This process is called the pozzolanic reaction and hydration (Rachman et al., 2018).

Conclusion

The control soil samples and tailings at the gold mine site in Kulon Progo are generally yellowish brown and contain sand, silt, clay, and a small amount of gravel. The initial mercury content in tailings has exceeded the environmental quality standard based on the 75 mg kg⁻¹ regulation by the Government of the Republic of Indonesia No. 22 of 2021. On the other hand, tailings contain mercury with the lowest concentration of 164.49 to the highest of 383.21 mg kg⁻¹, with the lowest pH being 6.74 to the most elevated pH being 8.56. The S/S method uses tras soil mixed with mercury-contaminated tailings; all specimen compositions meet the required minimum compressive strength value of 35 t m⁻² according to the US EPA and meet the TCLP value according to Government Regulation No. 22 of 2021 of 0.05 mg L⁻¹. The optimum value used in the tras soil and tailings mixture is the composition (90:10) of 90% tras soil and 10% tailings. The compressive test result is 31 t m⁻², and the TCLP test result is 0.0033 mg L⁻¹. In this study, the optimum conditions were selected based on using the most tras soil as a substitute for portland cement and passing the compressive strength test.

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