



# STABILIZATION/SOLIDIFICATION OF MERCURY CONTAMINATED SOIL OF TRADITIONAL GOLD MINING IN KULON PROGO YOGYAKARTA, INDONESIA USING A MIXTURE OF PORTLAND CEMENT AND TRAS SOIL

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## ABSTRACT

Traditional gold mining activity is one of the source of mercury contamination. Total mercury concentration around the tailing ponds in Kulon Progo ranged from 0.30 to 22.51 mg/kg, which the exceeded the quality standard values set by the government of Indonesia No. 101 of 2014 which is 0.3 mg/kg. One method that can be applied is stabilization/solidification (S/S). This study aims to determine the optimum composition of Portland cement and tras soil used in S/S with contaminated soil. The study at first step, Portland cement and tras soil compositions was used by variations of the weight value of 100: 0, 90:10, 80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 20:80, and 10:90. At second step, contaminated-soil samples were added into optimum from first step, with a variation composition of binder: soil iare 50:50, 40:60, 30:70, 20:80, and 10:90. Toxicity Characteristic Leaching Procedure was applied based on US EPA method 1311. First test result in optimum composition of Portland cement: tras soil (10:90), with the compressive test value of 96 kg/cm<sup>2</sup>. The second result of compressive test values of binder: contaminated soil (10:90) was 6 kg/cm<sup>2</sup> is with mercury concentrations in the TCLP test of 0.0011 mg/L. The result of compressive test met the quality standards of US EPA for the management of contaminated soil at 3.5 kg/cm<sup>2</sup>. The result of TCLP test met the quality standards for products of S/S in accordance with Government Regulation 101 Year 2014 at 0.05 mg/L

**Keywords:** mercury, Portland cement, stabilization/solidification, soil, tras.

## 1. INTRODUCTION

The activity of traditional gold mining in Kulon Progo Yogyakarta, Indonesia is operated conventionally in open locations using amalgamation technique i.e. mixing the sand powder with mercury to form amalgam (alloy) used as a gold binder (Rianto, 2010). Tailing waste as the result of mining activity is discharged into the underground and the river which make the soil and river mud contaminated with mercury. The result of mercury concentration analysis showed about 70-80% of mercury in the activity is lost into the river sediment, tailing, and ground (Mudyazhezha and Kanhukamwe, 2014). The result of previous study showed total mercury concentration around the tailing ponds in Kulon Progo ranged from 0.30 to 22.51 mg/kg, which the exceeded the quality standard values set by the government of Indonesia No. 101 of 2014 which is 0.3 mg/kg (Banunaek, 2016). Mercury that contaminates the soil, if the movement is not restricted, will go into the underground water which is the source of drinking water of the local communities (Wuana and Okieimen, 2011);

Mercury is identified as an element that has the quality of high toxicity and can be easily transformed into a more toxic form (Durube *et al.*, 2007; Rianto, 2010; Robles *et al.*, 2014). Mercury is dangerous to human life through the food chain. Mercury in the soil is absorbed by plants and it experiences a process of bio magnification (Wang *et al.*, 2012). Mercury one of the heavy metals contamination in the soil, which can be removed with remediation either biological, chemical, or physical methods.

One of remediation techniques that can be applied is the physical remediation technique are stabilization-solidification (S/S) (Naimoon, and Hamid, 2016). The technologies S/S may be applied to in situ or ex situ media. In situ processes treat soils in place, without removal and ex situ processes involve the removal of the contaminated media to a treatment area. This technology is often applied in the United States by the US EPA in the remediation of land contaminated with hazardous waste (US EPA, 2000). In general, the technology of S/S is an encapsulation process of waste into a solid material that involves mixing the waste with a binder to reduce the contaminant leach ability by physical and chemical means with low permeability which convert the waste into an environmental acceptable waste for safe disposal (US EPA, 1999; US EPA, 2000; US EPA 2001; US EPA 2004; Spence and Shi 2004).

In the process of S/S, common inorganic binder is used, among of them are: Portland cement, pozzolan, and a mixture of both (US EPA, 2006). Pozzolan is a material containing silica or alumina that has property for example, cement (US EPA, 2006). Portland cement is the hydraulic adhesive that reacts with water to bind other solids to form a unity mass of dense and hard (Intara *et al.*, 2011). Although Portland cement has the ability to properly bind contaminants in the solidification mechanism, the use of cement as a binding agent should be limited. In terms of its technical aspect, Portland cement is not proper for contaminant stabilization mechanism despite having excellent ability in the process of solidification. Therefore, to reduce the use and to improve the performance of Portland cement in the process of S/S, another additive



that also acts as a binding agent or stabilizing agent known as pozzolan is employed (Wilk, 2007).

Stabilization/Solidification using Tras soil that can improve the mechanical test of the product. Tras soil can also isolate heavy metals contained in it (Alina *et al.*, 2008). The result of study showed that Tras soil could potentially be used as a mortar mix with the indication of positive results on the test and durability of concrete produced. The addition of 50% of tras soil on the mixture of Portland cement - Tras soil within 28 days curing time increased the result test of concrete compressive by 1.6% (Intara *et al.*, 2011). Therefore, this research is aimed to determine the optimum composition of the mixture of Portland cement and tras soil for stabilizing the mercury contaminated soil in gold mining area in Kulon Progo, Yogyakarta.

## 2. MATERIALS AND METHODS

### 2.1. Material

Ordinary Portland Cement (OPC) and tras soil supplied from PT Semen Indonesia was used throughout this research. Soil sample as a control was taken from two control locations in the areas that are not contaminated with mercury and contaminated soil samples were taken at 5 locations at a distance of 10 meters from the spill tailings stream. Soil samples were taken at a depth of 30, Samples taken by digging the ground using a hand drill. Sampling activities were conducted at traditional gold mining, Kalirejo sub district, Kulon Progo, Indonesia. The activity was begin with a site survey to determine the sampling location based on the contour and the direction of water flow at the study location. The soil samples location can be seen in Figure-1.

### 2.2 Analysis

Each sample was then inserted into the plastic PET (Poly Ethylene Terephthalate) and labeled, and then inserted into ice box with a temperature of 4 °C and later brought to the laboratory for particle size analysis of soil and mercury analysis. Control soil and contaminated soil sampling was done by taking the soil at certain depths samplers method which refers to the US EPA (2001) on Methods for Collection, Storage, and Manipulation of Sediments for Chemical and Toxicological Analysis: Technical Manual. Contaminated soil samples from five locations were mixed into 1 composite sample. Likewise with the control soil samples from two locations are mixed into 1 composite sample.

All samples contained were later being analyzed of physical characterization of soils based on analysis of the distribution of particle-size refers to ASTM D422 including chemical characteristics of soil samples standard. Test Method for Particle-Size Analysis of Soils and chemical characteristics of soil samples conducted in Soil and Rock Mechanics Laboratory of Civil Engineering, FTSP ITS. Total mercury with mercury analyzer method. Mercury analyzer tool used was Lab Analyzer 254 Mercury Instrument Type VM-3000

Mercury Vapor Monitor. All samples were analyzed in double which later were separated as the end result.

The first step, Portland cement: tras soil compositions was used by variations of the weight value of 100: 0, 90:10, 80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 20:80, and 10:90. At second step, contaminated-soil samples were added into the optimum mixture of binder obtained from first step of study, with a variation of the weight value of binder: soil 50:50, 40:60, 30:70, 20:80, and 10:90.

Composition Portland cement and tras soil, variation of Portland cement and tras soil are shown in Table-1.

**Table-1.** Composition of portland cement and tras soil.

Sample	Portland cement (%)	Tras soil (%)
I	100	0
II	90	10
III	80	20
IV	70	30
V	60	40
VI	50	50
VII	40	60
VII	30	70
IX	20	80
X	10	90

The optimum Portland cement and tras soil which was obtained from the first composition with the addition of mercury contaminated soil. This first composition was performed in order to identify the optimum percentage of contaminated soil that can be added into the mixture of Portland cement and tras soil. Composition of optimum binding agent and the contaminated soil was shown in Table-2.

**Table-2.** Composition of optimum binding agent and contaminated soil.

Sample	Optimum binding agent (%)	Contaminated soil (%)
I	50	50
II	40	60
III	30	70
IV	20	80
V	10	90

### 2.3 Preparation of test objects

The test objects on the first composition were made by mixing tras soil with Portland cement. The production of the test object was done in several stages following USEPA method (US EPA, 2000).

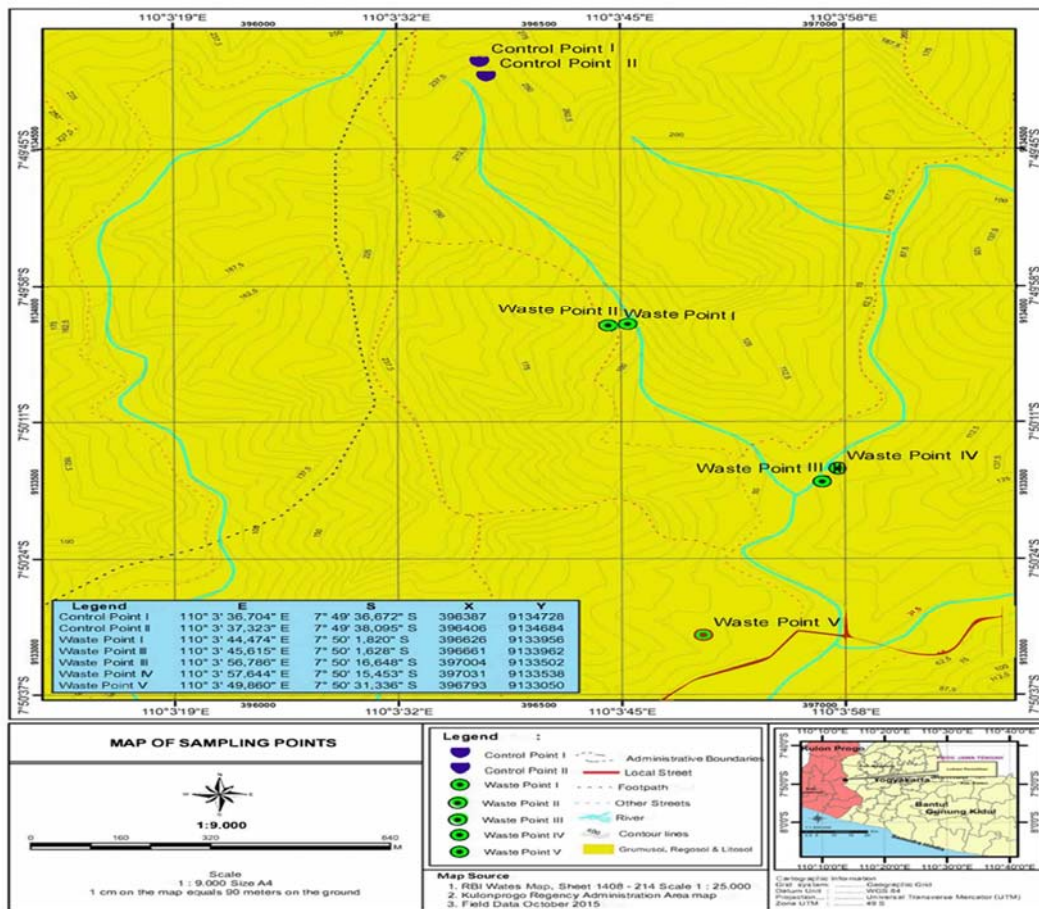


Figure-1. Map of soil sampling location.

### A. Preparation of soil sample

Preparation begins by heating the tras soil at 105°C oven for 24 hours. This activity was intended to eliminate the moisture content in tras in order not to disturb the grinding process. After that, the tras was placed at normal room temperature and then milled by bond ball mill with a speed of 56 rpm for  $\pm 3$  hours. Afterward, the tras was sieved using a sieve of 300  $\mu$ m in order to equalize its particle size.

### B. Normal consistency test

Normal consistency test aims to determine the water required in a mortar mixture. This test was performed using vicat Testing Machine. Normal consistency is reached when the vicat rod is placed on the surface of the mortar declines as deep as 10 mm at the least of 30 seconds. In normal consistency condition, the water evenly spread on the surface of the mortar from one end onto another end. The normal consistency test refers to ASTM C187-11 i.e. about Standard Test Method for Amount of Water Required for Normal Consistency of Hydraulic Cement Paste.

Normal consistency test was done by estimating the amount of water (distilled water) is added to a mixture of Portland cement and tras soil. As a starting point in

determining the volume of water added was a minimum of 35% of the mass of cement (Suparjo and Suhana, 2005).

After an unknown amount of water, then made the specimen using a cube-shaped mold (specimen molds) with a size of 5 cm x 5 cm x 5 cm. The mass of each specimen was 300 g. The test object is created the first phase of 10 units with two repetitions. In stage II made the test object as much as six with two repetitions. Total specimen made is 32 pieces.

### C. Mortar treatment (Curing)

The mortar treatment was done by keeping the mortar moist to prevent cracking in the test objects. Mortar was placed at room temperature for 28 days. In this study, moisture curing was performed, i.e. by placing the test objects around a bucket of water and then sealed using a large tub. This treatment aimed to minimize the leaching of heavy metals contained in Portland cement: tras mortar.

### D. Compressive strength test

A compressive strength test was performed as per ASTM C109-93 test method. The specimen molds with a size of 5 cm<sup>3</sup> after performed a compressive test after 28 days of curing. Compressive strengths of specimens were measure using Toorse Universal Testing Machine.



### E. TCLP (US EPA method 1331)

The TCLP test is performed to measure the leachable mercury contents in the course of this work, and determine the stability of treated solid wastes. Three pH measurements are recorded according to EPA Method 1311.

## 3. RESULTS AND DISCUSSIONS

### A. Characteristics of soil samples

#### a) Physical characteristics of soil samples

The soil samples of the mine site in the district Kalirejo, Kulon Progo, Indonesia were physically yellowish brown in color, dominated by sand, silt and clay. Physical characterization of soils is shown in Table-3 and Table-4.

**Table-3.** Analysis result of distribution grain of control soil samples.

Type of grain	Particle size (mm)	Content (%)	Water content (%)	pH
Sand		39.36	19.16	6.74
• Coarse	≥ 2.00			
• Medium	≥ 0.425			
• Fine	≥ 0.075			
Silt	≥ 0.0055	34.46		
Clay	≥ 0.0001	24.61		
Gravel	≥ 4.76	1.57		

**Table-4.** Analysis result of distribution grain of contaminated soil samples.

Type of grain	Particle size (mm)	Content (%)	Water content (%)	pH
Sand		55.17	13.24%	6.78
• Coarse	≥ 2.00			
• Medium	≥ 0.425			
• Fine	≥ 0.075			
Silt	≥ 0.0055	30.69		
Clay	≥ 0.0001	12.27		
Gravel	≥ 4.76	1.86		

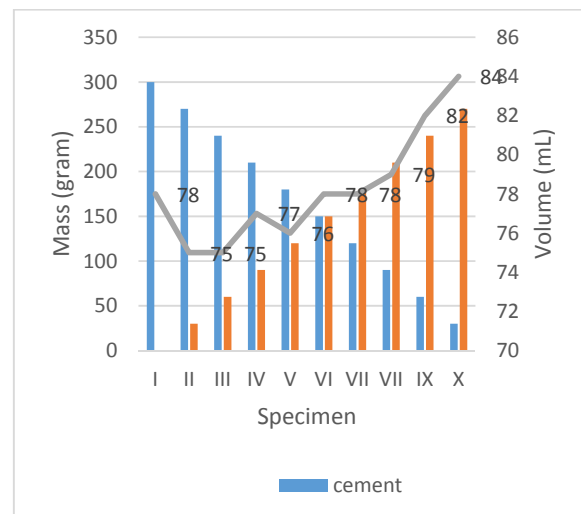
#### b) Chemical characteristics of soil samples

Chemical characterization of soil samples includes: pH and total mercury concentration. Soil samples were identified as having a relatively neutral pH valued at 6.74 for control soil and 6.78 for contaminated soil.

The result of total mercury level in control soil sample was 0.079 mg/kg. However, the result of total

mercury levels 0.89mg/kg in contaminated-soil samples was approximately ten times greater than the control soils. Mercury is a heavy metal that is toxic to humans and other organisms (Alpers and Hunerlanch, 2000). Under Regulation of 101/2014 value of quality standards of toxic characteristic through the TCLP and total concentration for the determination of the management of Hazardous Waste-contaminated soil is the total A concentration (TK-A) 300 mg/kg, TK-B at 75 mg/kg, and TK-C 0,3 mg/kg. Based on the quality standards, the soil of gold mining in Kulon Progo, Yogyakarta exceeded the quality standard of total C concentration (TK-C) so it can be said that the soil (land) have been contaminated with mercury as a result of gold mining process (Banunaek, 2016)

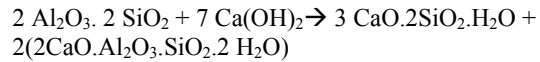
### B. The water requirement from the result of normal consistency test I



**Figure-2.** The graph of the consistency water requirement.

Based on the result of normal consistency test, it could be seen that with the increasing number of additions of tras to the mix cement, the water requirement tended to increase. In the cement content of 100%, the water requirement was 78 ml. Subsequently, when 10% and 20% of tras was added, the water requirement declined to 75 ml. The volume decline of this water need was caused by hydration reaction that was restrained. The reaction occurred at the beginning of the addition of water that produced a precipitate of  $\text{Ca(OH)}_2$ , ettringite, and C-S-H that formed coatings on the cement particles (Intara *et al.*, 2011). Consequently, the water requirement tended to increase with the addition of tras. Tras soil consists of silica-alumina reactive compound that is capable of reacting with water to form a new material into a solid mass unity (Indrawati and Manaf, 2008). This occurred due to the pozzolanic reaction between cement, tras and water (Intara *et al.*, 2011). The pozzolanic reaction that occurred is



**(a) The result of compressive test I**

Compressive test was calculated by the equation:  
Compressive Test ( $\text{kg}/\text{cm}^2$ ) = load/surface area. The result of the compressive test I is shown in Table-5.

**Table-5.** Test result of compressive test I.

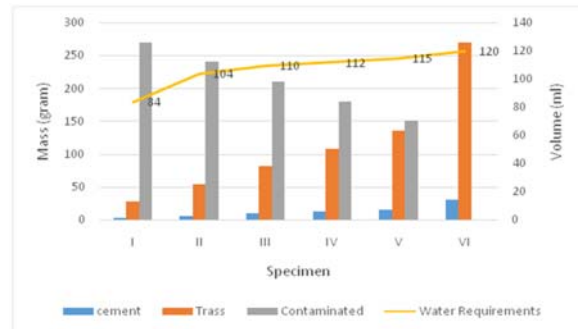
Specimen	Compressive load	The average		Compressive
	(kg)	compressive load		test
	A	B	(kg)	( $\text{kg}/\text{cm}^2$ )
I	12350	12000	12175	487
II	10750	8800	9775	391
III	10900	9600	10250	410
IV	9150	11000	10075	403
V	6550	9200	7875	315
VI	7550	8000	7775	311
VII	6500	4600	5550	222
VII	4900	5400	5150	206
IX	3550	5000	4275	171
X	1800	3000	2400	96

The value of compressive test on the test object I with a cement content of 100% was the highest. Next, the value of relative compressive test declined with the reduction of cement. In the test object III, however, the value of compressive test increased as much as  $19 \text{ kg}/\text{cm}^2$  of object test II. According to Widodo (2010) factors that affect the compressive test of concrete are: cement paste, cavity volume, aggregate, and the interface between the cement paste and the aggregate. In addition, another factor that affects the compressive test is technical factors, i.e. the imperfect manual printing and compaction process (Faisal, 2015). The increase of compressive test may occur due to the hydration process and the good interface between the cement paste and the aggregate.

Compressive test is one of the parameters for measuring the success of solidification process. Based on the parameters of the compressive test, all of the test objects met US EPA quality standards for the management of contaminated soil at  $3.5 \text{ kg}/\text{cm}^2$ . The determination of maximum composition was seen from the least use of cement with the consideration of economic side with the intention that this technology of S/S can be applied. With this consideration, it was determined the optimum composition of portland cement -tras mixture was on the variation of mixture of 10: 90.

**C. The water requirement of normal consistency test II**

The water requirement increased with the addition of contaminated soil. The increase of water requirement caused by the addition of the soil was because the soil samples with their physical properties of clay tended to hold water.

**Figure-3.** The graph of the consistency water requirement in test II.

According to (Intara *et al.*, 2011), the textured clay soil has a large specific surface area that the area has a high capability of water binding. When the water is bound by clay particles, it will stick so strong that the water can be retained in the clay particles. This was obviously seen in the test object II which had increased its water requirement compared to the test object I from 84 ml to 104 ml with the addition of 50% sample of contaminated soil.

**(b) The result of compressive test II**

The result of compressive test II is shown in Table-6. The result of the compressive test object I was the highest, which was  $96 \text{ kg}/\text{cm}^2$  without the addition of contaminated soil. Subsequently, the compressive test had decreased with the addition of contaminated soil.

**Table-6.** Test result of compressive test II.

Specimen	Compressive load (kg)		The average compressive load (kg)	Compressive test (kg/cm <sup>2</sup> )
	A	B		
I	1800	3000	2400	96.0
II	820	1040	930	37.2
III	560	520	540	21.6
IV	300	300	300	12.0
V	320	160	240	9.6
VI	140	160	150	6.0

This occurred because the levels of binder that acts as the glue mixture had decreased. The result of compressive test of the test object VI was the smallest with a composition of binder: soil (10: 90) which was 6 kg/cm<sup>2</sup>. The value still met the quality standards of compressive test of the product S/S according to US EPA by 3.5 kg/cm<sup>2</sup>.

#### (c) TCLP test

TCLP test was done referring to US EPA Method 1311 which is a parameter of the effectiveness of the stabilization process. TCLP test is one of the test requirements to determine the level of hazardous waste toxic in accordance with Government Regulation of 101/2014 hazardous waste. TCLP was done by crushing the test objects then extracted using the extraction solution in accordance with the pH of sample. The determination of the use of extraction solution was done by measuring the pH of initial sample and the pH of final sample. At the beginning of the samples measurement, the test objects were destroyed and distilled water was added. If the pH of measured sample is higher than 5 then 1 N HCl was added to it next the pH of sample was measured again as the final pH.

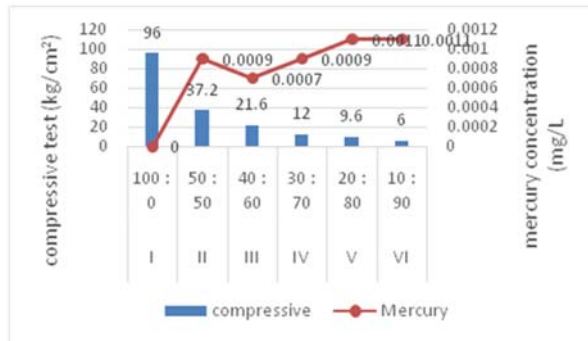
TCLP process is a simulation product S/S to the original environmental conditions at the site remediation (Suryono, 2011). Remediation of soil at the site contains organic acids that can potentially cause leaching of heavy metals which have been bound by the binder on the S/S. Therefore, TCLP extraction process performed using acetic acid buffer solution with pH  $\pm$  5. The result of Mercury levels in TCLP sample test is shown in Table-6.

**Table-7.** The result of mercury level.

Specimen	Composition binder: soil	Mercury concentration (mg/L)
I	100 : 0	Not detected
II	50 : 50	0.0009
III	40 : 60	0.0007
IV	30 : 70	0.0009
V	20 : 80	0.0011
VI	10 : 90	0.0011

From Table-7, the mercury concentrations increased with the addition of contaminated soil (BSN, 2011). The presence of mercury was not detected in the test object I with a composition content of 100%. Thus, the binder in the form of Portland cement-tras soil mixture was not contaminated by mercury. Mercury concentrations experienced fluctuation in the test object II, III, and IV and experienced increase in the test object V and VI. However, the fluctuation and the increase were not significant. It happened because of the low mobility of mercury at high pH.

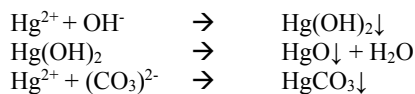
The value of mercury concentration in each test object is a parameter of the effectiveness of the stabilization process that occurs. In addition to the stabilization process, the value of the mercury in the test objects also has a correlation with the solidification process. This can be seen in Figure-4. Based on Figure-4, the mercury concentration was relatively increased with a decrease in the compressive test of the test objects. It occurred because the less binder used the less the value of the compressive test of the test objects that caused the mercury could not be tied to perfection. On the test object III, the concentrations of mercury decreased compared to the test object II with a larger binder composition. This was due to a manual technical manufacture of the test object, which was in the process of compacting and leveling of specimens in the mold. As a result, a mixture of the soil aggregate and the binder was uneven.



**Figure-4.** Relationship between compressive test and mercury concentration.

One of the factors that affect the test of mortar indicated by a value of compressive test is the interface of cement paste and aggregate (Widodo, 2010). Reduction in the compressive test shows the imperfect interface between binder and aggregate of contaminated soil. Therefore, when the TCLP extraction process occurs, there will be more mercury leached.

The decrease of mercury concentration in contaminated soil after S/S process was due to the process of stabilization of mercury in cement and pozzolan. Mercury will experience the sediment process forming a red precipitate  $\text{HgO}$  (Zhang *et al.*, 2009). The sedimentation occurred due to the reaction between  $\text{Hg}_2^+$  with alkali compound and carbon within pozzolan and cement. The reaction is written as follows:



The crystal structure formed from the hydration process is able to bind heavy metal contaminants after transforming to precipitated hydroxide and carbonate salt.

The test object VI with the highest levels of contaminated soil at 90% had the final concentration of mercury of 0.0011 mg/l. The value met the quality standards of mercury levels in accordance with Government Regulation of 101 of 2014 for the management of contaminated soil that is TCLP-B which is 0.02 mg/l. The optimum composition was selected based on economic and applicative considerations, i.e. the addition of the highest contaminated soil with the least consumption of binder. By this consideration, the optimal composition of stage II was in the composition variation of binder: soil (10:90).

#### 4. CONCLUSIONS

The optimum composition of binder Portland cement: tras soil from first study was 10: 90. This determination was based on the value of compressive test of the test objects which was 96 kg/cm². The optimum composition of the binder and the contaminated soil was 10: 90. At this composition, the compressive test of 6

kg/cm² was obtained and the total of mercury concentrations in the TCLP test was of 0.0011 mg/l.

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