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Stabilization/Solidification of Waste Containing Heavy Metals and Hydrocarbons Using OPC and Land Trass Cement

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ABSTRACT
The stabilization/solidification process (S/S) is one of the alternative methods of treating B3 waste, especially heavy metal. The S/S uses cement as the solidification agent. The cement will bind heavy metal pollutants in a monolithic mass with a sturdy structure, thus inhibiting its movement. The presence of hydrocarbons affects the S/S strength. Therefore, it is necessary to add pozzolan material which can absorb hydrocarbon constituting the cement blocking component of pozzolan cement, i.e. Ordinary Portland Cement (OPC) combined with trass soil. This study aims to determine the maximum content of organic materials in the form of hydrocarbons can stabilize/solidify heavy metals contained in wastes containing hydrocarbons. This research is conducted in two steps. Stage I aims to obtain the optimum composition of the mixture. On the other hand, stage II is to determine the maximum content of hydrocarbons in percent weight that can stabilize/solidify organic wastes containing heavy metals – Cu, Cr, and Pb in artificial wastes. The composition of OPC and trass soil was varied at a ratio of 100: 0, 5:25, 50:50, 25:75 and 0: 100. The hydrocarbons used in step II were paraffin, added to the optimum composition of OPC and trass soil with a proportion of 2.5%, 5%, 5% and 10%. The S/S product quality test was performed, involving: compressive strength test, Toxicity Characteristic Leaching Procedure (TCLP) and paint filter test. Strength test was conducted using a compressive strength testing apparatus Toasters Universal Testing Machine Type RAT-200, MFG No. 20380 CAP 200 tf. TCLP test under US-EPA (method 1311). The method of analysis pertaining to heavy metal concentrations involved a colorimetric method for Cr (VI), neocuproine for Cu, and dithizone for Pb. The paint test refers to the US EPA 9095B method. The results showed that the optimum composition of OPC mixture: trass soil was 50:50, which is the composition used in stage II. The results of compressive strength test were 2770 tons/m^2. The TCLP test results for heavy metals Cu and Pb with hydrocarbon addition on Cu 10% and Pb 2.5% reached 0.076 and 0.076 mg/L, respectively. The result of the paint filter test indicates that there is no remaining free fluid.

Keywords: trass soil, heavy metal, ordinary Portland cement (OPC), stabilization/solidification

INTRODUCTION

Environmental pollution by hazardous toxic waste (B3) is a serious problem that has not been handled properly. The B3 wastes generally contain the heavy metals resulting from industrial activities. The industrial waste containing heavy metals may be inorganic waste that does not contain hydrocarbons, for example including electroplating, metallurgical, and smelting industrial wastes.. The waste generated from the oil and gas industry can be liquid, solid/mud or gas. Large amounts of oil sludge is generated during tank cleaning, crude oil storage, maintenance of related facilities and processing activities prior to crude oil sales to sea terminals. Oil sludge contains aromatic hydrocarbons (benzene, toluene, ethyl benzene and xylene), poly-aromatic hydrocarbons and heavy metals (Ayotamuno et al, 2007).

Oil-borne waste is a water-in-oil emulsion residue (W/O) containing a hydrocarbon and heavy metal compound. The most commonly found compounds are petroleum hydrocarbons (PHC) compounds, ranging from 5% to 86.2%, and other organic compounds such as Polycyclic Aromatic Hydrocarbons (PAH), alkanes, and phenols. On the other hand, the content of heavy metals such as lead is 0.001–0.12 mg/kg, copper 32–120 mg/kg, and chromium – 27–80 mg/kg (Hu et al., 2013). Phenol and PAH compounds are flammable, so they are classified into hazardous and toxic waste or B3 waste (Silva et al., 2012). Oily mud contains organic and inorganic contaminants. The organic contaminants are in the form of heavy metals, such as zinc, lead, copper, nickel, chromium, and mercury. The organic contaminants consist of Total Petroleum Hydrocarbon (Al Futaisi et al., 2007). Many processing techniques are used for the processing of waste oil industry, especially oily sludge. The methods with oil sludge incineration have been introduced to replace the hoarding and bioremediation methods. However, the method has limited concerns that the combustion process can release toxic gases which evaporate into the environment. Inorganic vaporous (VOC) vapors are formed and released. Toxic heavy metals also cannot be removed during the combustion process and will accumulate as solid particles of combustion areas (Dominguez et al., 2005).

Petroleum wastes generated by businesses or activities of oil, gas and geothermal or other activities producing petroleum waste are hazardous and toxic waste. The waste has the potential to cause pollution and environmental damage. Therefore, it is necessary to manage it well. Technological alternatives have been developed for treating the B3 waste through the physical, chemical, biological or combinational mechanisms. The B3 waste treatment technology must be in accordance with the characteristics of waste. One of the methods of managing petroleum and soil waste contaminated by petroleum methods can be carried out by the biological treatment. However, the biological treatment has the requirements of Total Petroleum Hydrocarbon / TPH contaminant contents. The requirements of the biologically treated petroleum waste are the maximum concentration of the initial TPH before the biological treatment process is lesser than 15% (Decree of the Head of Environmental Impact Management Agency no. 128 of 2003). The results of the stabilization/solidification process can be reused for building materials, construction materials, road materials, or materials for pile up (Desogus et al., 2013). Cement is one of the most widely used binder materials in the S/S method (La Grega et al, 2005). According to Paria and Yuet (2006), the S/S waste technique using cement is one of the B3 waste treatment alternatives with the aim of reducing the environmental pollution. The S/S process is very effectively used in the treatment of radioactive waste and heavy metals. The S/S product is an immobilized heavy metal trapped in the binder (Yang and Min, 2008). The results of the S/S process can be reused for building materials, construction materials, road materials, or materials for pile up (Desogus et al., 2013). The S/S process using cement has been applied to the organic wastes containing hydrocarbons. However, the presence of hydrocarbons can reduce the formation of crystal structures, thus affecting the strength of the S/S products (La Grega et al., 2005). The organic substances in the waste do not form strong chemical bonds with cement. Therefore, the resulting S/S product has a poor compressive strength. (Karamalidis and Voudrias, 2007).

Some studies show that natural pozzolan is widely used as a substitute for the portland cement because it is cheaper, reduces heat, and increases the chemical resistance (Ghricia et al., 2007). The addition of pozzolan can reduce the amount of cement used in the S/S process. (Segui et al., 2013). Ezziane et al. (2007) stated that the addition of special pozzolan to the cement can increase the compressive strength of mortar. Zain et l.. (2010) conducted the S/S research for immobilizing the petroleum sludge. The proportion of the mixture was investigated to find the ratio of cement, rice husk ash, and sludge. Chaff ash (RHA) is added to 5, 10 and 15% cement replacement. The performance of S/S products is measured by compressive strength and permeable porosity. The results show that the addition of rice husk ash (RHA) 5% shows the best performance associated with free pressure at 24.9 N / mm^2. Porosity was found to increase along with the RHA content. The morphology of the compacted cement surface was found to be in the range of 10 to 15 µm for 15% RHA. The addition of ground granulated blast furnace slag (GGBS), pulverized fuel ash (PFA), MgO and Zeolite is an efficient combination in immobilizing the waste containing heavy metals and organic contaminants (Wang et al., 2015).

In ASTM C618, pozzolan is mentioned as a material containing silica and alumina, where the pozzolan material itself does not have binding properties such as cement. In its delicate form and in the presence of water, the compound reacts chemically at a normal temperature to form a binding-like compound such as cement or having a cementic ability. In ASTM C 593–82, it is argued that pozzolan is divided into two kinds, namely natural pozzolan and artificial pozzolan. The natural pozzolan is a natural material that is a pile or sedimentation material from the volcanic ash lava that which contains active silica. Trass is an example of natural pozzolan. Pozzolan is made from stoves and waste utilization products processed into ashes containing reactive silica through the combustion process, such as fly ash (ash), rice huck ash, and micro silica (silica fume). According to Borowski and Hycnar (2016), fly ash in granulated pattern with phosphogypsum constitutes a promising material in the production of cement, for the hardening of cement mortar.

In this research, trass soil was used as OPC mixing. This is because land trass is one of natural pozzolan banya available in Indonesia. Trass is also defined as the rocks from eruptions and volcanoes that have been weathered, gray-white, bluish-gray, dark-gray, yellowish, and somewhat orange (BPPI, 1983; BBPI 1984). Trass is found as a sediment layer with a thickness of 2–8 m or more. According to Susilorini (2003), the Muria Kudus trass can be utilized as an alternative aggregate in concrete mixtures. Laboratory tests show that the SiO_2 content of the Muria Kudus trass is 42.02%, while the Al_2O_3 content is 28.08%. Overall, concrete with a fine aggregate mixture of the Muria Kudus trass shows better performance than normal concrete. The result of compressive strength test is equal to 29,802 Mpa, which exceeds from compressive strength requirement (19 MPa). The trass soil is added because most of its content consists of silica, alumina, or both elements. The results of the study indicate that trass soil potentially serves as a mortar mixture with an indication of a positive result on the strength and durability of concrete produced. The addition of trass soil can increase the compressive strength (Alina et al., 2007).

However, the problem is whether trass soils can stabilize/solidify heavy metals Pb, Cr (VI) and Cu contained in the wastes containing organic substances. Thus, further research is needed to analyze the ability of trass soil with OPC mixture to stabilize/solidify heavy metals in organic waste and to what proportion of hydrocarbon can S/S products of the same quality as the applicable standards be generated. Besides, it is expected to absorb hydrocarbons to improve the process effectiveness. It is also expected to exploit the potential of trash land that is widely spread throughout Indonesia. Indonesia has a lot of trass soil potential spread, but still little utilization, as a substitute for some or all of the cement.

METHODS

The stages of research conducted in this study include preparation of tools and materials, manufacture, testing of S/S specimen products, and data analysis. The materials used are portland ordinary cement mixture with trass soil on various composition variations, and artificial wastes containing Cu, Cr (VI) and Pb heavy metals. A set of tools was used for the manufacture and testing of S/S specimens. Artificial wastes are made up of two types of waste, i.e., heavy-duty heavy metal waste without containing hydrocarbons (inorganic waste) and artificial heavy-metal wastes containing hydrocarbons (organic waste). Heavy-duty artificial metals used in accordance with unreacted waste will be discharged to Category I landfill, wherein the heavy metal content of Cu is higher or equal to 1000 mg/kg wastewater (dry weight), Cr is higher or equal to 2500 mg/kg (dry weight) os waste, and Pb is higher or equal to 3000 mg/kg (dry waste). As for the artificial wastes of heavy metals containing hydrocarbons, preparation carried out by adding liquid paraffin p.a. Preparation of artificial waste of Cu heavy metals is performed using $CuSO_4.5H_2O$, Cr heavy metals using $K_2Cr_2O_7$, and heavy metals Pb using $Pb(NO_3)_2$.

The study was conducted in two stages. In the first step, the process of making S/S specimens on inorganic waste types with mixed cement mixture of OPC and trass is 100: 0, 75:25, 50:50, 25:75, 0: 100. The specimen preparation begins with a Normal Consistency test. The test aims to determine the amount of water required in the OPC and trass cement mixture. Normal consistency test refers to ASTM C187–11. The specimen is printed in a cube mold called a 5×5×5 cm specimen mold, and cured for 28 days. Specimen preparation refers to ASTM C 109. Furthermore, the S/S product test, which includes compressive strength, Toxicity Characteristic Leaching Procedure (TCLP) test of compressive strength tested by Toce Universal Testing Machine Type RAT-200, MFG No. 20380 CAP 200 tf. TCLP test based on US-EPA (method 1311).

The analysis heavy metal concentrations was performed according to Standard Method for examination of water and wastewater, by colorimetric method for Cr (VI), neocuproine for Cu, and dithizone for Pb. Paint test refers to the US EPA 9095B method The optimum composition obtained in the first phase of the study is used in the second stage. The second stage is done on the type of organic waste containing hydrocarbons. The waste used is also an artificial waste of heavy metals Cu, Cr, and Pb containing hydrocarbons. Paraffins are added with composition variations in percent mass, ie 2.5%, 5%, 7.5%, and 10%. The S/S result product is also tested with the same test as the first phase of implementation. In this step, the paint filter test is conducted, which aims to find out the free liquid content remaining in the sample. The paint test refers to the US EPA 9095B method. The data obtained were analyzed statistically using One Way ANOVA (Analysis of variance) method with Minitab 16.

RESULT AND DISCUSSION

Compressive Strength Test

On the basis of the research results it was observed that the type of waste affects the value of compressive strength. The value of compressive strength on inorganic waste type without hydrocarbon enhancers ranges from 56 tons / m^2 to 295 tons / m^2. The highest compressive strength is exhibited by the sponge with OPC composition: 100: 0 trass soil of 6830 tons/m^2. Conversely, the minimum compressive strength value of 40 tons/m^2 is shown by the specimen with 100% trass soil composition. The results of the study on the type of waste without containing hydrocarbons indicate that all compressive strength values in all specimens still meet the minimum limit set by the Head Decision BAPEDAL N0. 03 year 1995 that is equal to 10 tons/m^2. The value of compressive strength on non-hydrocarbon inorganic waste types can be seen in Figure 1. Determination of the effect of the organic matter in the form of hydrocarbon on the compressive strength value is also done by using a statistical test. The test was conducted using ANOVA One Way method with Minitab 16. The analysis result showed a significance at α <0,05 or Fcount> Ftable which means that there is a significant difference in the composition % ratio. If the value of Fcount> Ftable then H0 is rejected and H1 is interpreted significant, it means there is a difference from the comparison group. Conversely, the value of Fcount <Ftable then H0 is accepted and H1 is not interpreted significantly, meaning there is no difference from the comparison group (Agus, 2013). The results of the statistical analysis showed that the value of Fcount> Ftable with P value of 1.57425. Thus, it can be interpreted as significant, which means that the composition ratio indeed has an effect on the value of compressive strength.

The value of compressive strength in the study of organic waste types with the addition of hydrocarbons can be seen in Figure 2. It shows the effect of adding hydrocarbons on the value of compressive strength. The more hydrocarbons are added, the lower the compressive strength. The more hydrocarbons in the form of paraffin added, the lower the compressive strength. This is because the cement cannot coalesce with the added hydrocarbon to form cavities in the stabilized/solidified specimens. The presence of cavities will result in differences in density, thus

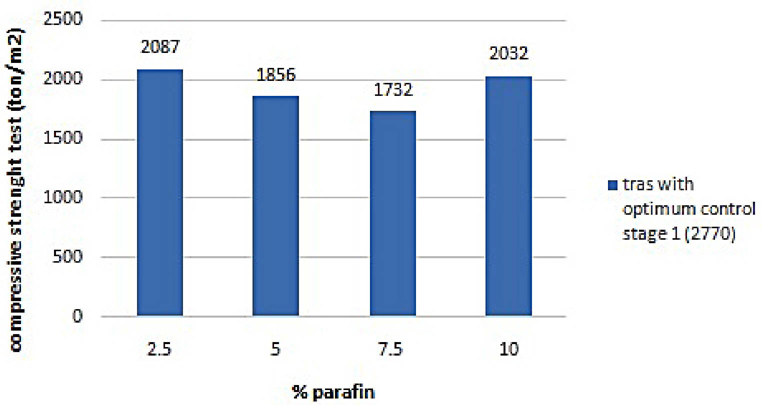

Figure 1 Strength Test Results of OPC and trass soil containing Heavy Metal (HV) without the addition of Hydrocarbons

Table. 1 ANOVA OPC Composition For Soil Trass Against Strong Press

Source of Variation	SS	Df	MS	F	P-value	F crit
Between Groups	4675008	1	4675008	0.23812	1.57425	4.964603
Within Groups	29696733	10	2969673			
Total	34371742	11				

affecting the density of the S/S specimen. The higher the density, the higher the value of the compressive strength. Leonard and Stegemann (2009) stated that the organic component does not match the cement matrix so that it can inhibit the hydration process. This hydration process is very important to determine the strength of cement. If the hydration of cement is disturbed, the compressive strength produced on cement becomes less than optimal.

TCLP Test Results

From the TCLP test results, leaching for heavy metals Pb, the best leaching was obtained on the OPC variation composition: trass soil 50:50 and 25:75, i.e. 0.114 and 0.105 mg/L. In the 75:25 variation of 0.12 mg/L, and on the 100% trass soil variations with the addition of heavy metals, the results still meet the quality standard (PP RI No 101 year 2014). This suggests that the S/S process with OPC variations and trass soils can immobilize Pb. Bajceta et l.. (2013) state that the S/S process can reach 99% Pb immobilization.

Leaching for Cu heavy metals with variations of OPC composition: best trass soil obtained at OPC composition: trass soil 25:75; 100% trass soil of 0.594 and 1 mg/L. In contrast, the lowest leaching was obtained in a variation of 100% composition of heavy metals, which amounted to 0.011 mg/L. The leaching results obtained between various composition variations indicate that the movement of Cu metal can be inhibited by the S/S process using OPC: trass soil. This is in accordance with research conducted by Bajceta et l.. (2013) that Pb, Cu can be immobilized up to 99% by using a modification of OPC and another pozzolan. The average value of heavy metal of S/S products with Tanah Trass can be seen in Figure 3.

In addition to the effects of heavy metal properties, curing time also affects the leaching from heavy metals. Pb and Cu result in lower leaching at curing for 28 days. However, Cr exhibits a higher leaching. The leaching of Cr started at 28 days of curing and the leaching effectiveness was achieved after 90 days of curing (Jain and Garg, 2008).

Determination of the influence of organic substances in the form of hydrocarbons on the value of compressive strength is also done by using statistical tests. The test was done by using ANOVA One Way method with Minitab 16. From the statistical analysis, it was found that in the table. 2 Fcount <Ftable with P value of 0.01021. so it can be interpreted as insignificant, which means that the composition ratio has no effect on leaching Pb. However, the results of leaching Pb still meet the quality standard. Table 3 showed from the results of statistical analysis that the value of Fcount <Ftable with P value of 0.03198. It can be interpreted as insignificant, which means that the composition ratio does not affect the leaching of Cu. However, Cu leaching results still meet the quality standards.

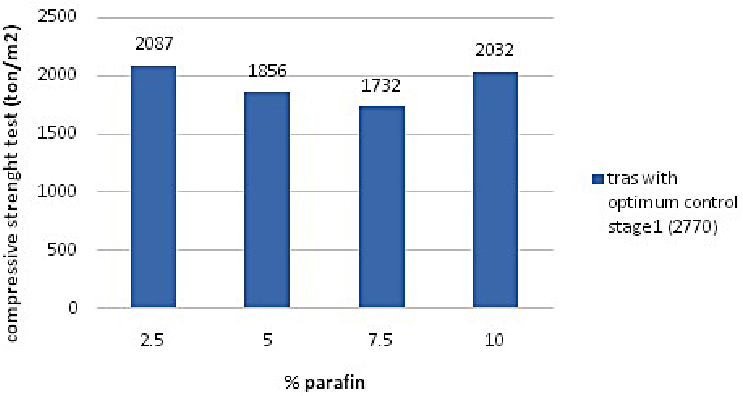

Figure 2 Strength test results of OPC and trass soil with the addition of hydrocarbons

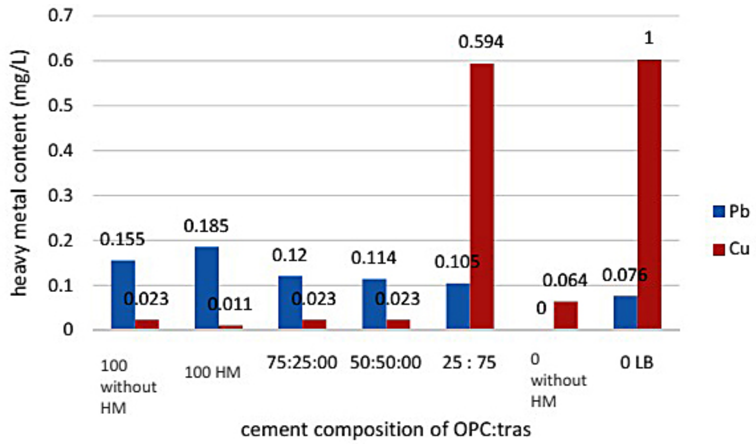

Figure. 3. Results of analysis Heavy Metal S / S Product OPC: Trass without the addition of Hydrocarbons

Table 2 ANOVA Composition of OPC For Land Trass Against Pb

Source of Variation	SS	Df	MS	F	P-value	F crit
Between Groups	8683.078	1	8683.078	9.261567	0.010211	4.747225
Within Groups	11250.47	12	937.5388			
Total	19933.54	13				

Table 3 ANOVA Composition of OPC For Land Trass Against Cu

Source of Variation	SS	Df	MS	F	P-value	F crit
Between Groups	5167.248	1	5167.248	6.200636	0.031984	4.964603
Within Groups	8333.417	10	833.3417			
Total	13500.66	11				

The TCLP test showed that the composition of OPC cement and 50:50 trass soil with paraffin addition, as shown in Figure 3, exhibits its unstable heavy metal leaching result on each heavy metal type. This is due to the presence of organic components that interfere with the cement hydration process, as well as the value of compressive strength.

Due to the presence of organic components, the hydration process does not take place perfectly, so the ability of cement and its mixture with trass soil, is less effective in stabilizing/solidifying Pb and Cu. The results of heavy metal piging of Pb and Cu are unstable, probably also the result of reading the absorbance value of the spectrophotometer. The absorbance readings occur with unstable numbers.

The addition of organic substances in the form of hydrocarbons, indicating the instability of heavy metals of TCLP test results. The first phase of the study showed that Pb leaching was 0.01 mg/L. whereas in the Phase II study, the Pb leaching became unstable in the 0.076 mg/L range with the addition of 2.5% paraffin, 0.107 mg/L at 5%, 0.076 at 7.5% and 0.104 mg/L at 10%. From statistic test using ANOVA one way method (Table 4), the P value equal to 0.368 indicated that the addition of paraffin on the specimen did not affect the leaching of Pb. This is because Pb leaching on each sample is not significant.

In the study without the addition of Cu hydrocarbon, only 0.02 mg/L leached. On the other hand, in the research with the addition of hydrocarbon, the leaching of Cu increased. The statistical test using ANOVA one way method (Table 5) with P value 0.02 value indicated that the leaching of Cu influenced the addition of hydrocarbon in the sample. This is in accordance with the statement of Minocha et al. (2003) that the organic component of the waste may interfere with the chemical stability of the S/S specimen.

On the basis of table 6, it can be seen that no free liquids are left in the sample so that all samples of S/S results pass the paint test. This shows that the addition of paraffin mass up to 10% of the

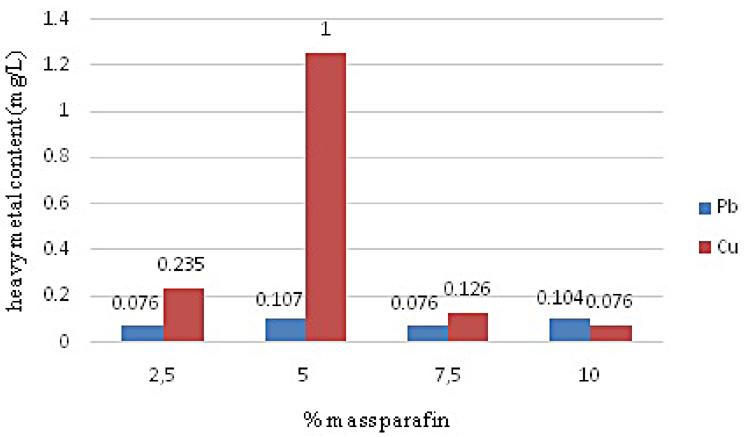

Figure 4. Results of heavy metal analysis of OPC 50: Trass 50 with the addition of hydrocarbons

Table 4 ANOVA% Paraffin For Trass Against Pb

Source of Variation	SS	Df	MS	F	P-value	F crit
Between Groups	82.23623	1	82.23623	26.31436	0.00684	7.708647
Within Groups	12.50058	4	3.125146			
Total	94.73681	5				

Table 5 ANOVA% Paraffin For Trass Against Cu

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	73.80131	1	73.80131	22.04824	0.009339	7.708647
Within Groups	13.38906	4	3.347265			
Total	87.19037	5				

Table 6. Hasil Paint *Filter Test*

% mass parafin	Name object of tested		Result of paint filter test
2,5%	H	H1	No detection of free liquid
		H2	No detection of free liquid
5%	I	I1	No detection of free liquid
		I2	No detection of free liquid
7,5%	J	J1	No detection of free liquid
		J2	No detection of free liquid
10%	K	K1	No detection of free liquid
		K2	No detection of free liquid

weight of the cement mixture of portland: trass soil can still be bonded to cement, although it affects the value of compressive strength and heavy metal leaching results.

CONCLUSION

This type of waste will affect the quality of the stabilization/solidification process. The effect of organic substances in the form of hydrocarbons on the quality of SS products is seen based on the value of compressive strength test and heavy metal lattice content after the TCLP test. In the maximum proportion of 10% hydrocarbon content, the value of compressive strength still far exceeds the standard quality standard, which is 1700 tons/m^2. However, with the presence of hydrocarbons, it can affect the TCLP test, which results in an unstable outcomes of the metal leaching. The results of the paint test showed that no stains were left in the sample.

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