Treatment of Lead Contaminated Soil using Solidification/Stabilization Method Incorporated with Sugarcane Bagasse

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In recent years, tremendous researches have been carried out for solid waste treatment using the solidification/stabilization (S/S) method incorporated with agricultural wastes after the incineration process. These researches, although they showed efficient results, but they may be expensive due to the incineration procedure cost. In the current research, the treatment of lead (Pb) contaminated soil was studied by the incorporation of sugarcane bagasse in its fibrous state into the S/S method. Chemical properties of the materials used were determined by X-Ray Fluorescence (XRF) test. Some mechanical tests like density, water absorption and compressive strength were conducted in order to meet the regulatory limits for disposing the treated waste. Some leaching tests were also conducted, to measure the leachability of lead (Pb) from the matrices. Solidification/stabilization was found as an effective method that was able to reduce more than 99% of leachability of Pb from polluted soil. Moreover, this method can incorporate until 10% of sugarcane bagasse into the matrices. Although incorporation of sugarcane bagasse up to 10% decreases the strength of the samples and increase the leachability of Pb, but they still fit to the standard. Incorporation of sugarcane bagasse waste in its fibrous state into the solidification/stabilization method may provide an alternative low cost treatment method for Pb polluted soils and may eliminate huge amounts of this waste from the environment.

Keywords: Solidification/stabilization, Lead, Sugarcane bagasse, Leachability, Compressive strength, solid waste, heavy metals, Agricultural wastes

The increase of wastes generation is becoming more and more obvious during the latest decades in Malaysia and in all over the world. Researchers have shown that between 40 to 80% of municipal solid waste is disposed of in developed countries whereas this rate reaches 60 to 90% in developing countries [1]. Several companies generate enormous quantities of residual substances that usually loaded by toxic heavy metals such as cadmium (Cd), copper (Cu), chromium (Cr), arsenic (As) and lead (Pb). In general, the heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders [2]. Mainly when these metals reach their contaminant threshold, they will become very harmful to both humans and the environment and persist in organisms when consumed [3]. Among these heavy metals, Pb considered highly toxic to plants and cumulative poison to the other beings [4]. It represents an important environmental and health issue all over the world, since it is very toxic and may cause disorders of nervous, reproductive and digestive systems [5]. The Pb contaminant in groundwater originates from the dissolution of Pb from soil as well as the earth's crust and from the combustion of leaded gasoline, which can contaminate the local surface water by surface runoff [6]. The United States Environmental Protection Agency (USEPA) has set the regulatory concentration limit for Pb at 5 ppm. So, up to this threshold, this heavy metal is considered very toxic [7].

Several methods and technologies have been developed for heavy metals treatment. These methods may include thermal and hydrometallurgical recovery [8], electrocoagulation [9], magnetic separation and purification [10], ion exchange electrodialysis [11], chemical precipitation [12], membrane filtration [13], coagulation [14], ûocculation [15], biosorptive flotation [16], encapsulation [17] and others.

Solidification/stabilization (S/S), which is considered very efficient, is one of the encapsulation methods that have been mainly used for the waste treatment. It has the role to immobilize the heavy metals [18]. This method is consisted of two operations: stabilization, which is a chemical method that attains to the chemical immobilization by the formation of stable compounds or water insoluble compounds [19], and the stabilization, which is the conversion of waste into a solid, hard and inert particulate material to encapsulate the contaminants within it.

Few years back, tremendous researches have been carried out for solid waste treatment using the S/S method incorporated with agricultural wastes ash [19-21]. These researches, although they have shown efficient results, but they have been resulted in expensive cost due to the incineration procedure to produce the ash. In fact, the only research that incorporated agricultural waste without incineration into the S/S method has been achieved by Janusa et al. in 2000 who have ensured the efficiency of

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SCB in Pb adsorption [22]. However, their research might not be useful for treatment of contaminated soils since they have used firstly the soaked SCB in a solution of water contaminated with Pb before blending it to the cement, while in reality the soil could not be separated from the contaminant before the treatment procedure. Moreover, their research has not identified the mechanical properties for the matrices formed [23-25].

The current research aims to determine the optimum percentage of SCB that could be incorporated into the S/S method that is used to treat the Pb contaminated soil as a sustainable low cost method. The characteristics of the different materials used were determined by XRF test to know the chemical compounds that may affect the properties of the matrices once formed. Some mechanical tests like density, water absorption and compressive strength were conducted in order to meet the regulatory limits for disposing the treated waste. TCLP and SPLP leaching tests were also conducted to measure the leachability of Pb from the matrices.

Experimetal part

Materials and methods

Soil samples

The clay was collected from the test site of the research center of soft soil (RECESS) in UTHM campus, Joho; then, it was dried in the oven and grinded to pass through the sieve of 300 µm. After that, the clay was blended with a precise amount of Pb $(NO_3)_2$ (which was dissolved in water before blending to ensure the homogeneity of soil) to reach a target concentration of 1000 ppm (this concentration was chosen in order to test the worst-case scenario of the contaminated soil) by using the following equation (eq. 1):

$$m = \frac{C * ms * Ms}{10^6 * \eta * n * Mm} \quad (1)$$

whereas:

m=	weight of the solution required	(g)
C=	target concentration of Pb	(ppm)
ms =	specimen final weight	(g)
n=	molecular number	
Ms=	solution molecular weight	(g/mol)
η=	percentage purity chemical	(%)
Mm=	metal atomic weight	(g/mol)

Cement and Water

Ordinary Portland cement type II (CEM II) was used as blender with distilled water.

Sugarcane bagasse waste (SCB)

SCB was collected from a small shop of sugarcane juice in Parit Raja, Johor. Then, it was treated with HCl by boiling it within 0.1M of the hydrochloric acid for 45 min. The boiling was repeated 3 to 4 times by changing the acid in each time until the filtrate was virtually colorless. This treatment was conducted in order to eliminate the maximum of the cellulose fibers and to liberate the lignin, because the presence of the soluble sugars, even at low concentration (0.03-0.15 wt.%) in cement may retard the setting time and the strength of this cement [22]. After treatment, SCB was dried under a temperature of 105 °C for around 24 h to eliminate the moisture content, then, it was grinded to pass over the sieve of 300 µm and saved in a plastic bag.

All the materials used have been preceded to the X-Ray Fluorescence (XRF) test in order to identify their chemical components.

Samples preparation

For the control samples, the contaminated soil was blended with 10, 15 and 20% of cement. For the other samples, the cement was replaced by 25 and 50% of SCB so that the role of SCB in these samples could be determined by comparison to the control samples. Water cement ratio (W/C) was ranged between 0.35 and 0.42 depended on the quantities of SCB added (the ratios have been determined experimentally because the fibrous nature of SCB may affect the matrices adhesion).

After specification of the materials' amounts, the samples were prepared one by one by blending soil, cement, SCB and water within a blender until the mixture was homogenous. Then, the samples were solidified one by one in a cylindrical mold, with the dimensions of 38 mm in diameter and 76 mm in height (38 mm x 76 mm). Within the mold, the confinement was in three layers within the mold by hitting almost 15 hits per layer, then; each layer was scratched to ensure its adhesion with the successive one. The solidified samples were then extracted from the mold and wrapped with a cellophane cover, then, they were left cured for 7 days and 28 days before conducting the mechanical tests. The curing was under a room temperature within a container that contains a few quantity of water to regulate the moisture within it.

Physical and mechanical tests

Unconfined compressive strength (UCS)

The test was carried out by using the Geocomp LoadTrac II system controlled by a software of calculation that is installed in a computer. The specimens were extruded from the molds, leveled, measured for length, diameter and weigh and placed in the center of the bottom platen of the loading device. Then, they were subjected to uniaxial compression test at a constant rate of strain of 1% per minute. The rate of strain was chosen so that the time to failure should not exceed about 15 min. The loading device was adjusted carefully so that the upper platen just made contact with the specimen. The load was applied to produce an axial strain at a rate of 0.5 to 2% per min. Load, deformation, and time values were recorded automatically to define the stress-strain graph. Loading was continued until failure of the specimen, then, the UCS values were recorded from the screen and saved [26].

Water adsorption test (WA)

This test was conducted according to ASTM C140 [27]. The test procedure was involved the drying of specimens in an oven under a temperature of 105°C until its weight was becoming constant, that weight was recorded. Then, the specimen was immersed in distilled water until its weight was becoming constant after excess water on its surfaces was wiped off using a dry cloth. The weight was also recorded. Water absorption percentage was estimated as the percentage of the ratio between the difference of dry and wet specimen and dry specimen (eq. 2):

$$W(\%) = \frac{Sw - Sd}{Sd} \quad (2)$$

whereas:

W =	water adsorption percentage	(%)
S_w =	weight of the wet sample	(g)
Sd =	weight of the dry sample	(g)

Density

Density values were calculated automatically by the LoadTrac II system from the UCS test.

Leaching tests

Toxicity Characteristic Leaching Procedure (TCLP)

This test is designed to simulate leaching in a municipal landfill by determining the mobility of heavy metals presented in the samples. The TCLP test was conducted according to USEPA Method 1311(modified). After the UCS test was carried out, 5g of the crashed samples were grinded to pass through 1mm standard sieve, then, the extraction procedure was carried out with a liquid to solid ratio of 20:1 in an acetate solution at *p*H 2.88 ± 0.05 in bottles and to be rotary agitated at 30 rpm for 18 hours using the rotary system (USEPA, 1992). After extraction in the acetic fluid, the solid and the liquid phases were separated by filtration through 0.45-mm-pore-size membrane filters. The pH was measured. After that, the filtrate was acidified with nitric acid to *p*H < 2, then, analyzed for metals using the atomic absorption spectroscopy (AAS). Then, the Pb quantity in the solutions obtained was compared with the regulatory cited by the USEPA which is 5 ppm so as to make decision about the efficiency of the procedure.

Synthetic Precipitation Leaching Procedure (SPLP)

This test, which simulates the acid rain, was conducted in this study according to the USEPA method 1312 (modified) (USEPA, 1992), where a mixture of dilute nitric and sulphuric (H_2SO_4/HNO_3) acids (*p*H of 4.20 ± 0.05) was used. Cautiously 60 g of concentrated sulfuric acid was mixed with 40 g of concentrated nitric acid 60:40. For this test, 5 g of each crushed specimen that passed over 1mm sieve was placed in a bottle container prior to add the leaching solution nitric/sulfuric acid (*p*H 4.20) to provide a ratio of 20:1 mass ratio of leachate to solidified specimens. After this, the mixtures were put in containers, then, agitated using a rotating extractor at 30 rpm for 18 h. Then, the extracts were separated by filtration through 0.45-mm-pore-size membrane filters. At the end of the extraction period, the filtrate was then acidified with nitric acid to *p*H < 2 and stored prior to AAS analysis for comparing with USEPA regulatory limit, which is 5 ppm. This procedure was made twice for all the samples to ensure the accuracy of the results.

Results and discussions

Characteristics of raw materials

Clay soil

Table 1 shows the chemical components of clay soil, which was mainly constituted of silica (61.30%), alumina (21.50%) and iron oxide (6. 61%) in major quantities and sulfur, potassium oxides and other elements in minor quantities. This result was supported by Saat et al. who had studied the clay from various locations in North-West Peninsular Malaysia and concluded that the major elemental contents of clay samples detected in the study were Si, Al, Fe, Ti and K [28]. The clay particles have negative net charges at their surfaces, which may be balanced by adsorbing cations [29]. Thus, the surface clay attracts cations presented in the water and this may give a clearer explanation to the colloidal property of clay.

According to ASTM C 618 [30] and Turkish Standard (TS 25) [31], the pozzolonic characteristics refer to the percentage of SiO_2 + AL_2O_3 + Fe_2O_3 ≥70%, which was fulfilled in this research where SiO_2 + Al_2O_3 + Fe_2O_3 = 89.41%. This result indicated the high pozzolonic characteristics of the clay. In the other hand, the clay sample contained a very minimal quantity of lime (CaO = 0.3), therefore, it cannot develop hydraulic properties, hence, hydrated lime or material that can release it during its hydration, such as Portland cement, is then required to activate the natural pozzolans as a binding [32].

Table 1
CHEMICAL COMPONENTS OF CLAY SOIL

Formula	Concentration
Orig-g	7
Added-g	3
CO_2	0.1 %
SiO_2	**61.30 %**
Al_2O_3	**21.50 %**
Fe_2O_3	**6.61 %**
SO_3	3.99 %
K_2O	2.92 %
MgO	1.35 %
TiO_2	1.08 %
NaO_2	0.41 %
CaO	0.30 %
Cl	0.12 %

Cement

Table 2 shows the percentages of different compounds of cement (CEMII). It is clear that the highest percentage was the lime (CaO = 56.50 %), followed by the silicate (SiO_2 = 15%) and aluminum, sulfur, iron and potassium oxides and other elements in lower percentages. The presence of lime in the cement may activate the natural pozzolans when it is added to the clay as a binder material and increase its strength.

Table 2
CHEMICAL COMPONENTS OF CEMII

Formula	Concentration
Orig-g	8
Added-g	2
Cao	**56.7 %**
SiO_2	**14.8 %**
C	10.00 %
Al_2O_3	3.89 %
SO_3	3.07 %
Fe_2O_3	2.60 %
K_2O	0.81 %
MgO	0.57 %
SrO	0.26 %
TiO_2	0.19 %
Na_2O	0.18 %

Sugarcane bagasse

Table 3 shows the chemical properties of the different components of SCB. In this table, Fe_2O_3, SiO_2, Al_2O_3 and Cl were presented in major quantities, which were 3.16%, 1.15% and 3.23% respectively. The low concentration of these elements may indicate that SCB contains very low pozzolonic characteristics. Due to this matter, SCB could

Table 3
CHEMICAL COMPONENTS OF SUGARCANE BAGASSE (SCB)

Formula	Concentration
Orig-g	8
Added-g	2
C	0.10 %
Cl	**3.23 %**
Fe_2O_3	**3.16 %**
SiO_2	**1.82 %**
Al_2O_3	1.15 %
Cr_2O_3	0.71 %
S	Low Limit Detection
CaO	0.22 %
MoO_3	0.22 %

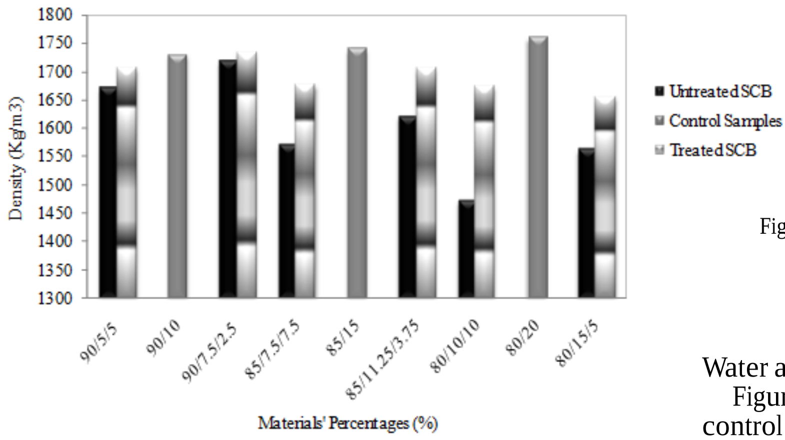

Fig. 1. Density of Samples after 28 Curing Days

not be a replacement for the cement. On the other hand, the high percentage of Cl is simply due to HCl treatment of SCB.

Mechanical properties

Density

Figure 1 shows the density values in function of different percentages used after 28 curing days. In the control samples (0% SCB), the increase of density was insignificant and equal to 1730 kg/m^3, 1740 kg/m^3 and 1760 kg/m^3 for 10, 15 and 20% of cement, respectively. Nevertheless, the incorporation of 3.75, 7.5 and 10% of SCB decreased the density of samples until 7%, 10% and 16%, which are clearly explained by the low density of SCB itself. This low-density may provide important advantages, such as, easier handling of wastes, and low transport cost [33].

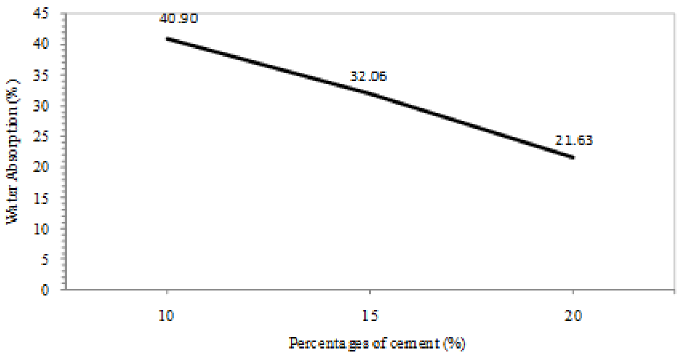

Fig. 2. Water absorption for Control Samples

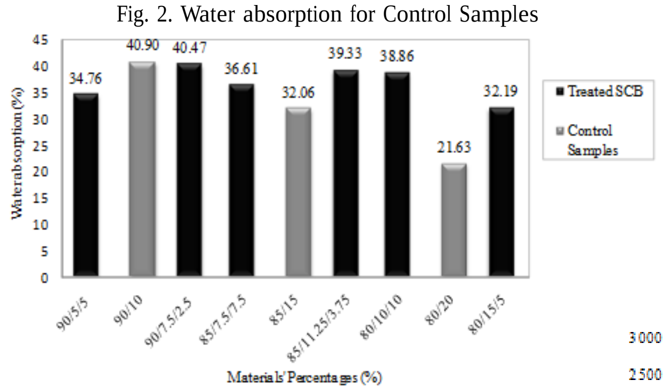

Fig. 3. Amounts of water absorbed by the different samples

Water absorption (WA)

Figure 2 shows the amounts of water absorbed by the control samples (0% SCB). The water absorption (WA) decreased almost linearly with the increase of cement percentages from 40.9 for 10% of cement to 21.6 for 20% of cement. This decrease may be due to the increase of strength in function of cement percentages so that the samples became less porous.

Figure 3 shows the water absorbed by samples after adding SCB. It were observed that the incorporation of SCB increased the amount of WA for all the samples except the sample of 5% of SCB with 5% of cement and the sample of 7.5% of SCB with 2.5 of cement where the change was not clear due to the few quantity of SCB added. The increase in WA might be due to the ability of SCB as fibrous material to absorb the water using the capillarity force. However, the high amount of WA may lead to the destruction of the matrices if the quantity of cement is not sufficient to maintain the matrices in their solidified forms. This result was also discussed in the UCS test part.

Unconfined Compressive strength (UCS)

According to the British Standard (BS), the limit strength for solid waste to be disposed in the landfill is 340 kPa. Due to this matter, the UCS value of such sample has to exceed this limit before disposing, so as to prevent its destruction. Figure 4 and figure 5 show the UCS for 7 and 28 curing days.

Control samples

For the control samples, it was observed that the USC increased throughout the time, and the lowest strength values obtained were 1374 kPa and 1798 kPa for 7 and 28 days respectively with 10% of cement. These results, which may refer to the role of curing time in the solidification of the matrices, are explained by the water that still existed within the samples, and evaporated through the time. Hence, these samples need enough time to complete the evaporation and increase the UCS values [34].

In addition, these results exceeded the landfill disposal limit even after only 7 curing days. Thus, the S/S method is efficient with the use of only 10% of cement as low cost percentage in this research. Moreover, the UCS was

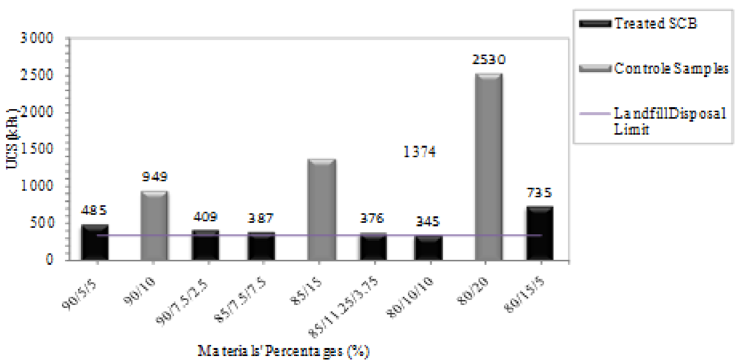

Fig. 4. Compressive Strength after 7 Curing Days

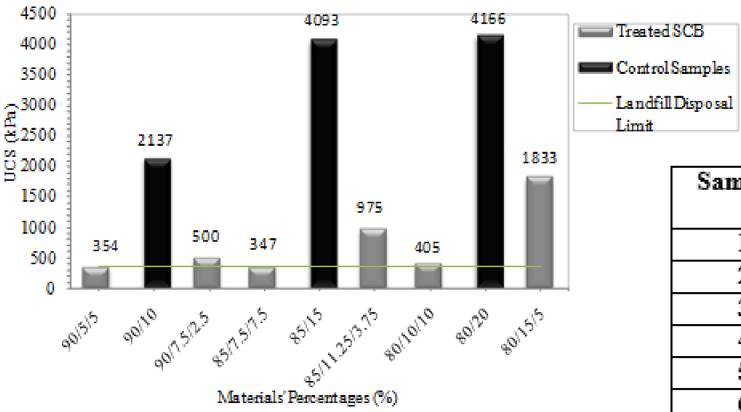

Fig. 5. Compressive Strength after 28 Curing Days

increased in function of cement percentages. However, after 28 curing days, the UCS were 2137 kPa, 4023 kPa and 4166 kPa for 10, 15 and 20% of cement, respectively, and this may be explained by the increase of lime percentage in the matrices, which plays its role as an activation of natural pozzolans [32].

Incorporation of SCB

The incorporation of SCB although it decreased the UCS values compared to the control samples but they still fit to the standard and exceeded the landfill disposal limit for all the samples, which is 340 kPa. In this case, the lowest strength obtained was 345 kPa in blending 10% of SCB with 10% of cement for only 7 curing days. Moreover, after 28 curing days, it is clear that the UCS decreased with the increase of SCB percentage. These results may refer to the low pozzolonic characteristics of SCB as described in the XRF results.

These results also can support the one obtained in the WA test, which indicated that the incorporation of SCB increased the amounts of water absorbed by the samples, hence, SCB affected the states of the solid samples and lead to the decrease of the UCS values.

From the UCS test, an alternative sustainable solution to eliminate the SCB from the environment is to incorporate it up 10%, into the S/S matrices, but this result should comply the leaching results for final decision of its efficiency.

Leaching tests

In combination with the mechanical properties tests, with the results of the leaching tests a decision for the efficiency of the S/S method could be taken, based on the USEPA regulatory limits.

Toxicity characteristic Leaching Procedure (TCLP)

After the extraction process, the filtrates were subjected to the AAS analysis.

The Pb was not detected for all the control samples by the AAS machine, which conducts to the efficiency of the S/S method even with only 10% of cement. This result may be explained by the formation of Pb $(OH)_2$, which is insoluble, thus, the Pb is captured within the matrices, and is prevented to be leached out. The following equation (eq. 4) explains the chemical reaction within the matrices.

$$Pb(NO_3)_2 + CaO + 3H_2O = Pb(OH)_2 + 2HNO_3 + Ca(OH)_2 \quad (4)$$

After one repetition of the leaching, the AAS showed very near results for some samples and compatible results for the others, so that the ultimate values were the average of the two results in each percentage.

Table 4
TCLP RESULTS OF THE INCORPORATION OF SCB

Samples	Mixture percentages (%)	Pb Concentration (ppm)
1	$S_{90}C_5SCB_5$	7.4
2	$S_{90}C_{7.5}SCB_{2.5}$	5.3
3	$S_{85}C_{7.5}SCB_{7.5}$	4.5
4	$S_{85}C_{11.25}SCB_{3.75}$	0.8
5	$S_{80}C_{10}SCB_{10}$	2.4
6	$S_{80}C_{15}SCB_5$	0.0

Table 4 shows the TCLP results of the incorporation of SCB. It is observed that the Pb was detected at very low concentration for all the samples, which were below 5 ppm, except for the sample of 5% of SCB with 5% of cement and the sample of 2.5% of SCB with 7.5% of cement, where the concentrations exceeded the threshold and were detected at 7.4 ppm and 5.3 ppm, respectively. After solidification, the maximum concentration of Pb was 7.4ppm, which was negligible compared to the concentration before solidification (1000ppm): 7.4/1000 =0.0074< 0.01. This means that the S/S matrices retained more than 99% of the Pb. Referring to UCS results, it is observed that the samples of 3.75% of SCB with 11.25% of cement and 5% of SCB with 15% of cement that correspond to the highest UCS values, which were 975 kPa and 1833 kPa, respectively, showed minimal leaching of Pb which were 0.8 ppm and 0.0 ppm, respectively. Otherwise, the sample of 5% of SCB with 5% of cement that correspond to a low value of UCS, which was 354 kPa, showed the highest leaching of Pb, which is 7.4 ppm. These results are explained by the role of cement and strength of samples in maintaining the Pb and prevent it to leach out from the matrices.

Synthetic Precipitation Leaching Procedure (SPLP)

For the SPLP test, no leaching for Pb was recorded for all the samples (with and without SCB). This finding is excellent demonstrates the efficiency of the S/S method against the acid rain.

Conclusions

In this study, solidification/stabilization (S/S) method incorporated with sugarcane bagasse (SCB) was used to eliminate the lead (Pb) from contaminated clay soil. As a result, this method was able to reduce the leachability of Pb more than 99% from contaminated soil. In addition, the optimum proportions of the S/S method were 10% of SCB incorporated with 10% of cement. Although incorporation of SCB decreased the UCS of samples but they complied with the standard. This result may provide an alternative sustainable low cost treatment for the S/S method and eliminate huge amount of SCB from the environment. Based on these results, treatment of real contaminated soil should be conducted in future researches to ensure the efficiency of this method. Moreover, other types of agricultural wastes may be incorporated with the S/S method in order to test their abilities that may provide an extra sustainable solution towards the environment.

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