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THE EFFECT OF CEMENT AND RICE HUSK ASH ON THE COMPRESSIVE STRENGTH AND LEACHABILITY OF ARTIFICIALLY CONTAMINATED STABILIZED SEDIMENT

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ABSTRACT

Solidification/Stabilization is an effective, yet economic remediation technology to immobilize heavy metals in contaminated soils, sediments and sludges. The main objective of this research was to study the effect of replacing cement with rice husk ash (RHA) on compressive strength and leachability of Pb from the stabilised sediments. Cement and Rice husk ash were used as stabilizing agents to immobilise lead contaminated sediment. In this study, artificially contaminated sediment was prepared by spiking $Pb(NO_3)_2$ to achieve an average of 1000 ppm target concentration to bracket a worst case scenario. The Pb- impacted sediment was stabilized with 10% cement and 5, 10, 15 and 20% rice husk ash (RHA) by total dry weight of the mixture and was cured for 7, 14 and 28 days. The unconfined compressive strength test (UCS) and the toxicity characteristic leaching procedure (TCLP) was used to evaluate the effectiveness of the S/S treatments at 7, 14 and 28 days. The result of the Toxicity characteristic leaching procedure (TCLP) leaching test conducted on the lead spiked solidified samples indicated that with addition of 15% RHA at 7, and 14 days the Pb concentration was below the leachability limit of 5 mg/l, subsequent 28 days of curing, the concentration of lead was all below the leachability limits except the control sample. The effect of pH on the leachability of lead (Pb) was also considered in the study, the leachability of lead in the TCLP at 7, 14 and 28 days respectively decreases from 13.49 mg/l to 1.89 mg/l as the pH of the leachates increases from 3.57 - 5.13. It was also observed that the higher the strength of the solidified sample the lower the leaching rate of lead in the TCLP. The X-ray diffraction (XRD) results indicated that lead in $Pb(NO_3)_2$ had precipitated to form $Pb(OH)_2$ within the cement hydration environment which explained the high treatment efficiency due to low solubility of the precipitates. Results have indicated that the partial replacement of cement with RHA in the binder system has increased the strength and reduced leachability of Lead from the treated sediment samples compared to the untreated ones.

Keywords: lead, strength, rice husk ash, TCLP, spiked sediment, X- ray diffraction, immobilisation.

INTRODUCTION

Lead (Pb) is one of the most common contaminants found in soils and sediments as a result of agricultural activities, urban activities and industrial activities such as mining and smelting. Lead (Pb)-based compounds have been a major source of environmental contamination in the past few decades (Tang and Yang, 2012). The major health impacts of the lead include anaemia, rise in blood pressure, brain damage, miscarriages, central nervous system (CNS), kidney and sperm damage. Lead can have detrimental effects upon hemoglobin production and kidney function (Bradl, 2004). The remediation technology of solidification/stabilization (S/S) offers an effective means of treating the lead-contaminated sediments by considerably reducing the mobility and solubility of lead in the sediments. Most of the research work carried out on S/S of metal-contaminated sediments was mainly focused on the use of ordinary Portland cement (OPC) or incorporated with other pozzolans such as fly ash and lime, but very little research has been done on the use of rice husk ash with cement for the treatment of lead (Pb) contaminated sediments. Rice husk is produced in millions of tons per year as a waste material in agricultural and industrial processes. After burning rice husk, the rice husk ash (RHA) produced as a by-product, about 20% of its original weight (Anwar *et al*., 2000, Chindaprasirt and Jaturapitakkal, 2009). It is one of the most suitable source of pozzolanic material among agricultural waste as it contains a relatively large amount of silica. Rice husk ash is a highly pozzolanic material; it contains non-crystalline silica and high specific surface area that are accountable for its high pozzolanic reactivity (Chindaprasirt and Rukzon, 2008; De Sensale, 2006). The high silica content in the form of non-crystalline or amorphous silica of RHA is dependent on the burning temperatures; 95% silica could be produced after heating at 700°C for 6 hours (Della and Hotza, 2002). Several research works have been conducted on the use of rice husk ash as mineral additive to improve the performance of concrete [Zhang *et al*, 1996, Coutinho, 2003 and Isaiah *et al;* 2003). However, its application in hazardous waste treatment is relatively new and is under investigations. The use of RHA and cement has been studied in stabilizing lead contaminated soil (Yin *et al.*, 2006). Results have indicated that usage of OPC with RHA as an overall binder system for S/S of lead-contaminated soils is more favorable in reducing the leachability of lead from the treated samples than a binder system with standalone OPC. Much of the work on solidification/stabilisation of metals in soil/sediments focused mainly on using cement alone or with other pozzolans, but very little research has been done on the use of rice husk ash with cement for the treatment of dissolved lead (Pb) in sediments.The objective of this study was to investigate the effects of replacing cement with RHA on the compressive strength, and leachability of lead contaminated sediment.

MATERIALS AND METHODS

Materials

Sediment sample was collected from Sembrong river at a site located between geographical coordinates of latitude 1° 52'.18.44" N and longitude 103° 06' 15.71" E, at Parit Sempadan, Parit Raja near Universiti Tun Hussein Onn Malaysia (UTHM). Sediment samples were then preserved in clean plastic bags, transported to the laboratory and kept in the tray for air drying until further analysis.

The rice husk used was obtained from milling of rice at Muar, it was burnt at a controlled temperature of 700ºC in the furnace for a period of 6 hours, with a heating rate of 5°C /min and then it was left overnight to cool. The burnt ash was then grounded using Ball mill so as to produce ash of size less than 75μm. The pozzolanic reaction of RHA obtained under controlled incineration conditions is far superior to any of the known pozzolanic materials, burning rice husks at temperature of 700 °C produces rice husk ashes with high pozzolanic activity (Ramezanianpour *et al*; 1995, Ramezanianpour and Rahim, 2000, and Vosugh 2001).

The Cement used is Holcim Top standard cement (CEM II/B - M32.5 N). This type of cement is usually used as the main binder for ordinary construction and is readily available. The cement for this study was added at a constant amount based on total dry weight of the mixture at 0% and 10%.

Sediment contamination by spiking

Initial analysis of the sediment indicated that it contained low concentrations of heavy metals which is below detection limits. The sediment was subsequently spiked with a known quantity of reagent grade lead (II) nitrate $(PbNO_3)_2$. The nitrate form of Pb was chosen due to its high solubility, representing a "worst-case" scenario (Dermatas and Meng, 2003), and to the fact that it does not inhibit S/S reactions (Yin *et al*., 2006). The spiked sediment was prepared by mixing a predetermined amount of lead(II) nitrate (98% purity) to obtained approximately 1000 ppm target concentration with deionised water prior to addition into the sediment. The sediment was mixed thoroughly to ensure homogeneous distribution of the contaminant in the sediment. After being thoroughly homogenized, the Pb spiked sample was allowed to mellow for a period of 7 days

Solidification/Stabilization sample preparation

The spiked sediment sample was mixed with the required amount of cement and/or RHA. Although based on current practice, cement has been added to soil from 5% to 20%, it was decided to experiment with 10% cement and rice husk ash was added from 5% to 20% of the total dry weight of the mixture. The sediment, cement and RHA were then thoroughly mixed in a clean Baker stand mixer. The water cement ratio used was 0.4 for all the mixing process. The total mixing process took approximately 7 minutes, recommended by EuroSoilStab (2002).

The mixture was then transferred to a custom-made plastic cylindrical mould of 25mm diameter and 50mm height and compacted in 3 layers of 10g each. Each layer was compressed and compacted with a custom made miniature compaction tool and compressed for 25 blows at each layer. The sample specimen was then gently pushed out of the mould and wrapped with a cling film and kept at relative humidity of not less than 95% in tightly closed plastic curing box at 27 ± 2°C for different periods of up to one month. UCS test was carried out on the specimens at 7, 14 and 28 days of curing. A detail of the mix ratios of the specimens for the unconfined compressive strength (UCS) test is given in Table-1.

Table-1. Mix Ratios for unconfined compression Test (Cement + RHA + spiked sediments).

Mix Ratios symbol	Sediment (%)	Cement (%)	RHA (%)	w/c ratio
A	100	0	0	0.4
B	90	10	0	0.4
C	85	10	5	0.4
D	80	10	10	0.4
E	75	10	15	0.4
F	70	10	20	0.4

Notation:
A = 100:0:0 denotes 100% Spiked sediments, 0% Cement and 0% RHA
B = 90:10:0 denotes 90% Spiked Sediments, 10% Cement and 0% RHA
C = 85:10:5 denotes 85% Spiked Sediments, 10% Cement and 5% RHA
D = 80:10:10 denotes 80% Spiked Sediments, 10% Cement and 10% RHA
E = 75:10:15 denotes 75% Spiked Sediments, 10% Cement and 15% RHA
F = 70:10:20 denotes 70% Spiked Sediments, 10% Cement and 20% RHA

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Unconfined compressive strength test (UCS)

The unconfined compressive strength (UCS) test was conducted to evaluate the strength of the solidified/stabilized sediment samples. The test was conducted according to BS1377:1990: part 7 [BS 1377-7 (1990)] and was carried out using the strength testing machine (LoadTrac II Geocomp, USA). Each specimen used in the UCS was compacted in a cylindrical plastic mould, 25mm in diameter by 50mm height (25 x 50 mm). This test was conducted on the solidified samples to indicate whether the treated material had adequate strength to support any overburden pressure. Specimen tested for UCS were collected and dried in the oven at 105°C for 24 hours before being crushed to pass through a 1 mm sieve prior to TCLP test.

Toxicity characteristics leaching procedure test (TCLP)

The TCLP test was conducted in accordance with EPA Method 1311 to evaluate the leachability of Pb from the Pb-spiked stabilized sediment. All samples were passed through a No. 40 sieve (0.425mm) prior to the TCLP tests. Precisely, 5g of the crushed stabilised sediment which was further grinded using Ball mill to fine particles passing 425μm was placed in 500-ml plastic bottles and mixed with a designated 100mL of TCLP extraction fluid. The appropriate extraction fluid for all mixtures was determined based on the pH of the spiked sediments as specified in the procedure. The extraction fluid used was acetic acid (pH 2.88 ± 0.05), at a solid-liquid ratio of 1:20 by weight of the crushed stabilised sediment. All samples were agitated using End - over - End rotating extractor at 30 rpm for 18 hours at room temperature in accordance with the procedure. The pH of the leachates was each measured at the end of the extraction period, and the leachate was filtered through a 0.45 μm pore-size membrane filter. To avoid metal precipitation the leachate pH was reduced to less than 2.0 after filtration by adding few drops of nitric acid before the metal analysis, because metals remain dissolved in pH less than 2.0.

X-ray diffraction (XRD) analysis

XRD was used to investigate the crystalline mineral phases responsible for Lead $(PbNO_3)_2$ immobilisation in the stabilised sediment samples. Representative samples which are Pb28d.100:0:0 (Untreated sample) and Pb28d.70:10:20 (Treated sample) were air dried for 24hr and then were ground to pass through a US standard No. 200 sieve (75μm). Step-scanned X-ray diffraction data was collected with XRD Bruker D8 (Germany).The XRD analyses were conducted at 40 kV and 40mA using a diffracted beam graphite-monochromator with Cu radiation. The data were collected over the range from 10° to 90° with a step size of 0.02° and a count time of 15.4 s per step. XRD patterns were qualitatively analysed and interpreted using Eva software and reference data base from the International Centre for Diffraction Data (ICDD) data base.

RESULTS AND DISCUSSIONS

UCS of stabilised/solidified lead spiked sediment sample

Compressive strength is one of the criteria used to judge the quality of the solidified material. The results for unconfined compressive strength (UCS) data of the Lead spiked solidified cylindrical samples are presented in Table-2. It could be seen that as the ratio of the RHA increases the compressive strength also increases as the number of days of curing increases (7, 14 and 28 days) except the control samples Pb100:0:0 (100% lead spiked sediments, 0 % cement and 0% RHA) at 14 and 28 days having the strength of 246 kPa and 236 kPa respectively. Increase in the RHA quantity (5 - 20%) enhances the compressive strength of the solidified material. Maximum compressive strength (7493 kpa) was obtained for sediment mix ratio of Pb28.70:10:20 (70% spiked sediments, 10% cement and 20% RHA) at 28 days curing period and the lowest compressive strength was obtained for sample Pb28.100:0:0 (100% lead spiked sediment, 0% Cement and 0% RHA) of 236 kPa at 28 days curing period. The results showed that the strength increases with respect to curing time. The high strength development was observed from sample containing 20 % RHA during the 28 days of curing. This is caused by the calcium hydroxide that was produced during cement hydration and which increased to the level that is enough to dissolve silica from RHA. As a result, the secondary calcium silicate hydrate generated from reaction between calcium hydroxide and soluble silica from RHA is the cause of additional strength gain in cement blended with RHA (Asavapisit *et al*., 2001, Asavapisit *et al*., 2005, Asavapisit and Ruengit, 2005). It was observed that UCS values of all the solidified/stabilised samples (except for the control samples at 14 and 28 days) exceeded the minimum Landfill disposal limit of 0.34 N/mm^2 (340 kPa).

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Table-2. UCS of Solidified/Stabilised Lead spiked samples throughout 28 days of curing.

Mix ratios	Unconfined compressive strength (Kpa)		
	7 days	14 days	28 days
100:0:0	371	246.5	235.8
90:10:0	2102	2875	3975
85:10:5	2218	3383	3426
80:10:10	2135	2541	4887
75:10:15	2606	4522	4561
70:10:20	2787	3549	7493

Leachability

Table-3 shows the different concentrations of lead (Pb) that was leached out in the TCLP extraction at 7, 14 and 28 days, and the efficiency of treatment carried out. As can be seen the highest leaching concentration of 13.49 mg/l of Pb was obtained for the control sample (Pb7. 100:0:0) at 7 days while the least lead concentration of lead in the TCLP observed was 1.890 mg/l at 28 days for the treated sample (Pb28d. 70:10:20). The treatment efficiency of 86.4% (11.6% leached out) is the specimen with high leachability and 98.37% (1.63% leached out) is the specimen with lowest leachability of lead indicating high treatment efficiency. The result of the TCLP leaching test conducted on the lead spiked solidified samples indicated that due to addition of 15% RHA at 7, and 14 days the Pb concentration was below the leachability limit of 5 mg/l, subsequent 28 days of curing, the concentration of lead was all below the leachability limits except the control sample which was not treated. The high treatment efficiency may be attributed to the change of pH of the treated sediments as indicated by the pH of the leachates in Figures 1, 2 and 3. It was expected that the mechanism responsible for the effective treatment was hydroxide precipitation. As a result of the increase in pH of the treated sediments, the lead was retained in the form of insoluble hydroxide within the solidified matrix (LaGrega, 2001 and Conner, 1990.

Table-3. Concentration of lead (mg/l) in the TCLP leachate.

Mix ratios	Conc. at 7 days (mg/l)	Conc. at 14 days (mg/l)	Conc. at 28 days (mg/l)
100:0:0	13.49	10.03	13.09
90:10:0	6.391	6.604	3.594
85:10:5	6.290	6.082	3.920
80:10:10	5.835	5.307	3.610
75:10:15	3.769	3.722	2.624
70:10:20	2.977	2.912	1.890

Relationship between strength and leachability of lead spiked sediment samples

The leachability of lead in the solidified matrix is related to the strength of the stabilised sediment sample, as can be seen in table 4. The highest leaching rates of lead obtained in the TCLP, were 13.49 mg/l, 10.03mg/l and 13.09 mg/l at 7, 14 and 28 days respectively when their corresponding compressive strength were 371 kPa, 247 kPa and 236 kPa which were very low compared to the treated samples with different binder additions. The lowest leaching concentrations in the TCLP of 2.977 mg/l, 2.912 mg/l and 1.89 mg/l was also obtained at 7, 14 and 28 days respectively with the strength of 2787 kPa, 3549 kPa and 7493 kPa at 7, 14 and 28 days respectively. So from this we can conclude that the higher the strength of the solidified sample the lower the leaching rate of lead in the TCLP. The untreated samples have high leaching rate compared to the treated sample. The reason been that it is well known that the calcium silicate hydrate (C-S-H) addition of RHA to the cement, it chemically reacts to the CH to produce additional C-S-H gel which contributes to improve microscopic property of cement (Dakroury and Gasser, 2008). The production of more C-S-H gel in concrete with RHA may improve the concrete properties due to the reaction among RHA and calcium hydroxide in hydrating cement (Yu *et al.,* 1999).

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Table-4. Relationship between strength and leachability of lead in the TCLP at 7, 14 and 28 days of curing.

Mix ratios	UCS 7days (kPa)	TCLP 7days (mg/l)	UCS 14days (kPa)	TCLP 14days (mg/l)	UCS 28days (kPa)	TCLP 28days (mg/l)
100:00:00	371	13.49	246.5	10.03	235.8	13.09
90:10:00	2102	6.391	2875	6.604	3975	3.594
85:10:15	2218	6.29	3383	6.082	3426	3.92
80:10:10	2135	5.835	2541	5.307	4887	3.61
75:10:15	2606	3.769	4522	3.722	4561	2.624
70:10:20	2787	2.977	3549	2.912	7493	1.89

Effect of pH on the leachability of lead (Pb)

The results showed that the pH of the lead spiked sediment increased after stabilization. This may be attributed to the dissolution of calcium, sodium and potassium hydroxides in the cement. It can be seen from Figures 1 - 3 that the leachability of lead in the TCLP at 7, 14 and 28 days respectively decreases from 13.49 mg/l to 1.89 mg/l as the pH of the leachates increases from 3.57 - 5.13. The leaching of lead was observed to be lower after stabilization for the different rates of cement and rice husk ash added. Appel and Ma (2002) reported an increase in Pb sorption with the increase in soil pH, they observed that increase in one unit of pH in a range of 2 to 7 resulted in 28% increase in Pb sorption. The solubility of metals is known to be lowered at higher pH values (Inbaraj and Sulochana, 2002).

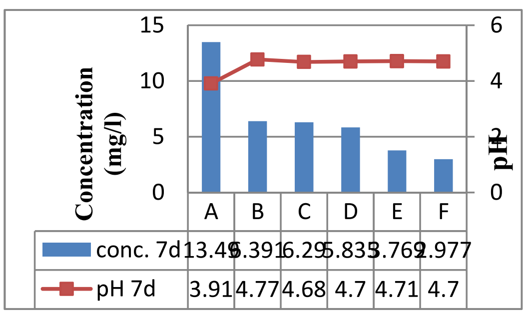

Figure-1. The effect of pH on the leachability of lead in the TCLP at 7 days.

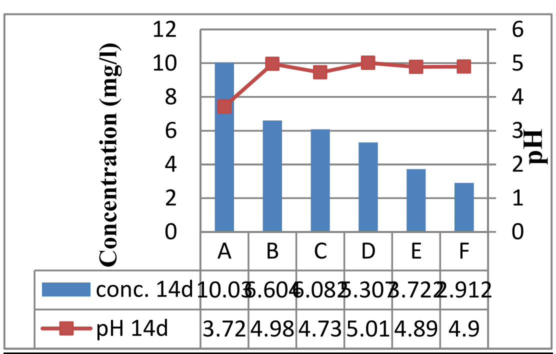

Figure-2. The effect of pH on the leachability of lead in the TCLP at 14 days.

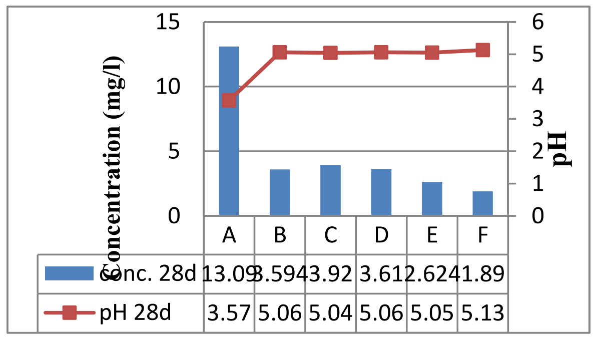

Figure-3. The effect of pH on the leachability of lead in the TCLP at 28 days.

XRD of lead spiked sediment

The results of the X-ray diffractograms evaluation shows the formation of various phases of Cement as well as RHA stabilized sediments after 28 days of curing for the various crystalline phases. Figure-4 shows the XRD patterns of the control and treated Lead spiked sediments sample after 28 days of curing (Pb28.100:0:0 and Pb28.70:10:20).

The most prominent peaks in the pattern of the untreated lead spiked sediment as can be seen in Figure 4 were those of Quartz (SiO_2) at 2θ of 20.87°, 26.36°, 36.56°,50.12°, and 59.96°, pyrite (FeS) at 2θ of 33.02°, 40.75°, 47.56° and 56.25°, Kaolinite at 2θ of 12.36° and 24.92°. There were distinct peaks of $Pb(NO_3)_2$ at 2θ of 19.53°, 32.34°, and 38.14°. Following the treatment with cement and RHA (Pb28d.70:10:20) there were no distinct peaks of $Pb(NO_3)_2$. The presence of lead hydroxide ($Pb(OH)_2$) peaks at 2θ of 24.92°, 27.23° and 28.29° also indicated that lead in $Pb(NO_3)_2$ had precipitated to form $Pb(OH)_2$ within the cement hydration environment which explained the high treatment efficiency due to low solubility of the precipitates. This observation supports the finding of a study conducted by Li *et al.* (2001) who proposed that heavy metals such as lead could exist in the S/S matrix as metal hydrated phases or metal hydroxides precipitating on the surface of calcium silicate hydrate (CSH). The peaks of ettringite were observed at 2θ 15.78°, 22.96° and 40.93°. Portlandite $Ca(OH)_2$, one of the cement hydration products, was identified at 2θ of 18.11° and 50.78° which indicated that pozzolanic reactions were not over by the end of the 28-curing day period.

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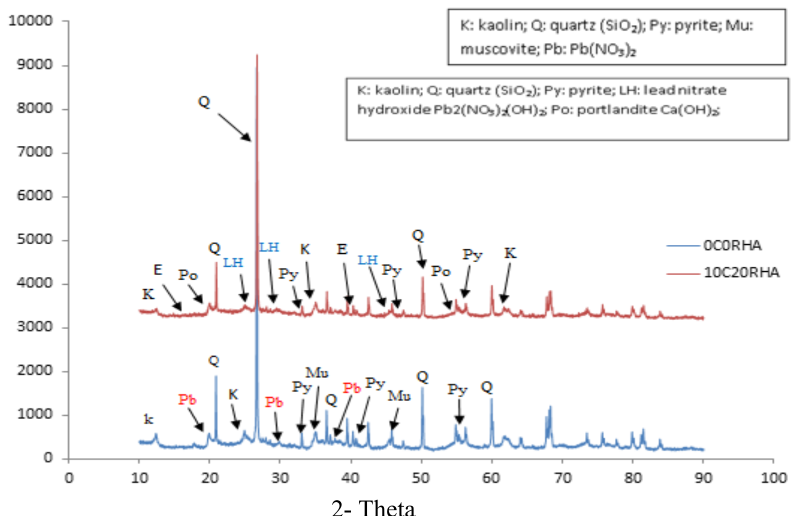

Figure-4. XRD patterns of Lead spiked sediment before and after treatment subsequent 28 days. Notation: 0C0RHA denotes 100% Lead spiked sediments, 0% cement, 0% RHA at 28 days curing period. 10C20RHA denotes 70% Lead spiked sediments, 10% cement, and 20% RHA at 28 days curing period.

CONCLUSIONS

The following conclusions can be drawn based on the experimental results obtained of this study

The unconfined compressive strength (UCS) for the lead spiked sediment after 28 days of curing days was in the range of 246.5 to 7493 kPa. There was enormous increase in the compressive strength with increase in the rice husk ash content of 5 - 20%, as the number of days of curing increases. The maximum strength obtained was between 15-20% RHA additions. The minimum strength was obtained for the control samples (100% sediment, 0%cement and 0%RHA). All the UCS values obtained for the solidified sediment samples exceeded the Landfill disposal limit of 0.34 Nmm^2 (340 kPa) for a waste, except for their control samples which are slightly below the regulatory limit.

a) Leachability test (TCLP) conducted on the crushed stabilized/solidified lead spiked sediment sample indicated good treatment efficiency. Results for the TCLP indicate addition of (15%) show high retention capacity of lead (Pb) compared to the control sample.

b) The immobilization of $Pb(NO_3)_2$ was achieved due to the precipitation of lead nitrate to form lead hydroxide $Pb(OH)_2$ in the cement hydration which explained high treatment efficiency due to low solubility of the precipitates. Pozzolanic reactions after 28 days of curing were also observed not to be over as residual portlandite $Ca(OH)_2$ was identified for the treated sample.

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