The relationship between unconfined compressive strength and leachate concentration of stabilised contaminated sediment

Mohammed Kabir Aliyu 1*, Ahmad Tarmizi Abd Karim1 and Chee–Ming Chan1

1Faculty of Civil and Environmental Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Johor, Malaysia

*Email: gf150243@siswa.uthm.edu.my

Abstract. Solidification/Stabilization (S/S) treatment was used in this study to immobilise copper (Cu) in contaminated river sediment. The sediment was artificially contaminated by spiking the solution of Copper sulphate (CuSO₄·5H₂O) to so as to get an average of 1000 ppm target concentration. Portland composite cement and Rice husk ash (RHA) were used as S/S agents. The amount of cement added to the mixture was 10% and while rice husk ash at the rate of 5%, 10%, 15% and 20% to the total dry weight of the mixture and then was cured for 7, 14 and 28 days. The unconfined compressive strength test (UCS) and toxicity characteristic leaching procedure (TCLP) were used to evaluate the effectiveness of the S/S treatments. From the results obtained it indicates that the partial replacement of cement with RHA in the binder system has increased the strength and the leachate concentration of copper was less in the treated sediment samples if compared with the untreated ones.

Keywords: sediment, rice husk ash, stabilisation, copper

1. Introduction

Solidification/stabilization (S/S) treatment is a technology whereby waste materials are treated in a way that changes the physical and chemical properties of the contaminants. Thereby decreases their spread via leaching thus reducing the threat they pose to the environment. The process might include chemical bonding or physical entrapment of the hazardous mixtures. Most applications of S/S either employ Ordinary Portland Cement as the only binder or may be mixed with other ingredients such as lime, fly ash, blast furnace slag etc [1]. Due to the fast industrial development in the last few years huge loads of contaminants are added to our rivers [2]. Out of these contaminants, heavy metals are of major concern because of their persistent and bio-accumulative nature. One of the concerned environmental problems these days is contaminated sediments; the existence of aquatic organisms on and inside sediments makes them significant for their well-being and health. If sediments become contaminated, they can pose a threat to sediment dwelling habitats [3].

Compressive strength is one of the standards used to judge the quality of the solidified material. The strength of the mixture varied depending on the additives added. Compressive strength is the most valuable property of concrete, even though in many practical cases other characteristics, such as durability, impermeability and volume stability, may in fact be more important [4]. The calcium silicate hydrate (C-S-H) addition of rice husk ash (RHA) to the fresh cement, it chemically reacts to the CH to produce additional C-S-H gel which adds to improve microscopic property of cement [5]. 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 [6].

However, incorporation of RHA as partial replacement of cement increases the compressive strength
of concrete but the optimal replacement level by RHA to give full long term strength improvement has
been reported between 10% up to 30% [7]. The RHA contains a very high amount of silica content and
which is found to be amorphous in nature. The amorphous silica contained in RHA can react with
cementitious binders to perform pozzolanic activity [8]. RHA is a highly pozzolanic material; it contains
non-crystalline silica and high specific surface area that are accountable for its high pozzolanic reactivity.
Numerous research works have been conducted on the usage of rice husk ash as mineral additive to
improve the performance of concrete [9-11]. However, its application in hazardous waste treatment is
relatively new and is under investigations. The objective of this study is to examine the effects of
substituting cement with rice husk ash (RHA) on the compressive strength, and leachability behaviour of
Cu in the stabilised sediment.

2. Materials and Methods

2.1 Materials used

2.1.1 Sediment

Sediment sample was collected from Sembrong river, Batu Pahat, Johore, 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) as shown in Figure 1. The equipment used
to take out the sample from the river was sediment core sampler, (Beeker sampler, Netherlands) as shown
in Figure 2. The sampler consists of a transparent tube of 1 or 1.5 metres, piston, piston rod and battery
powered pressure and vacuum pumps with extension hose. The advantage of sediment core sampler is
that it is fast and reliable, there is no need to assemble and disassemble tool with each new sample. The
sediment samples were collected at a depth of around 4-5 metres from the surface of the river.

Figure 1. The location where sediment samples were taken (1° 52'.18.44" N and 103° 06'15.71" E)
2.1.2 Cement
The Cement used in this study is Holcim Top standard cement and this kind of cement is commonly used as the main binder for normal construction and is easily obtainable. The cement for this study was added at a constant amount of 10% base on the total dry weight of the mixture.

2.1.3 Rice husk ash
In this study, the rice husk used was burnt at a controlled temperature of 700°C in the furnace for a period of 6 hours, with a heating degree of 5°C /min and was at that moment left overnight to cool. This is a controlled burning process. Then after that the burnt ash was then grounded using Ball mill grinder so as to produce an ash of size less than 75µm as seen in Figure 3. The ash was kept in an airtight clean plastic before usage. The pozzolanic reaction obtained of rice husk ash (RHA) burnt under regulated incineration conditions is far better to any of the known pozzolanic materials, thus burning rice husks at temperature of 700°C produces rice husk ashes with high pozzolanic activity [12-13].

2.2 Sediment contamination by spiking
Initial analysis of the sediment showed that it contained a very low concentration of toxic heavy metals below the detection limits; therefore the sediment was subsequently spiked with a known quantity of copper sulphate (CuSO₄·5H₂O). The salt of copper sulphate was chosen due to its high solubility in water. The spiked sediment was prepared by mixing a predetermined amount of Copper sulphate (99% purity) to obtained approximately 1000 ppm target concentration with deionised water prior to addition into the
sediment. The target concentration of 1000 ppm was chosen to simulate worst case contamination scenario of copper in the landfill. The sediment was mixed thoroughly to ensure homogeneous distribution of the contaminant in the sediment. After being thoroughly homogenized, the Cu spiked sample was allowed to mellow for a period of 7 days so that the contaminant will attached very well with the soil particles.

2.3 Binder system
After the mellowing period of the spiked contaminant (Cu), the treatment of the Copper - spiked sediment was carried out using cement as the main binder and augmented by adding rice husk ash (RHA) at dosages of 5, 10, 15 and 20 wt%, while the cement added at constant amount of 10% to the total dry weight of the mixture all through the mixing processes. Recent study on the use of RHA as a construction material has been reported by Jayasankar et al. [14], Nargale et al. [15] and Sandesh et al. [16], where the amount of replacement varies from 0 to 20% without varying the grade of ordinary Portland cement (OPC).

2.4 Unconfined compressive strength (UCS) test
Unconfined compressive strength test (UCS) was conducted to assess the strength of the mixture of rice husk ash with cement and sediment samples at higher stress. It is an important parameter in soil mechanics and plays a vital part when dealing with engineering decision for any soil. The unconfined compression test was carried out in accordance with BS 1377 (1990), part 7. It was carried out using the strength testing machine (LoadTrac II Geocomp, USA) as shown in Figure 4.

![Figure 4. Unconfined compression test machine (LoadTrac II, Geocomp, USA)](image)

The samples for UCS determination were prepared in triplet and each specimen was compacted in a cylindrical mould, 25mm in diameter by 50mm height. The compaction of the mixture was carried out using a specially designed miniature compacting tool at three layers with 30 blows each.

The specimens were gently extruded from the moulds, leveled, measured for length and diameter. Care was taken to ensure that both ends of the specimen were as flat as possible to curtail bedding error during tests, especially with firmer specimens. Then the specimen was placed between the top and lower platens as shown in (Figure 5). An axial load was then applied vertically at a constant rate of strain of 1mm per minute. According to the standard, failure generally indicates the condition in which the specimen can withstand no further increase in stress. i.e. the point at which it offers its full resistance to deformation in terms of axial stress. The unconfined compressive strength (q_u) is taken as the peak stress of the soil stress-strain curve or, if in cases where the axial stress does not readily reach a maximum value, failure is deemed to have occurred when a 20% ( i.e. equivalent to 15.2 mm for a 76 mm specimen) axial strain has
been reached. The compressive strength of the solidified samples was determined after 7, 14 and 28 days of curing. The Samples 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 leaching test.

![Figure 5. Solidified specimen placed between the top and lower platens](image)

2.5 Toxicity characteristics leaching procedure (TCLP) test (Method 1311)
The TCLP test was conducted in accordance with EPA Method 1311 to assess the leachability of Cu from the stabilized Cu-spiked sediment. All samples were passed through a No. 40 sieve (0.425mm) prior to the TCLP tests. Specifically, 5g of the crushed stabilised sediment which was further grinded using Ball mill to fine particles passing 425μm was put in the 500-ml plastic bottles and was mixed with a selected 100ml of TCLP extraction fluid. The suitable extraction fluid for all mixtures was determined based on the pH of the spiked sediments as stated 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. Then all the samples were agitated using End - over - End rotating extractor at 30 rpm for 18 hours at room temperature in accordance with the TCLP method.

3. Results and Discussion

3.1 Unconfined compressive strength test of the stabilised sediment
The compressive strength tests of the stabilised sediment samples were carried out at the end of each curing periods (7, 14 and 28 days). Table 1 shows the results for the compressive strength of the stabilised sediment and Figure 6 also illustrates the effect of RHA addition on compressive strength for different mix ratios and curing periods. As we can observe higher compressive strength was obtained at an early age of 7 days especially with 5, 10 and 15% RHA addition but the level of strength improvement decreases at 14 and 28 days. The maximum strength (4522 kPa) was obtained at 7 days using 15% RHA addition, and the lowest compressive strength (292.1 kPa) was obtained with the control sample (100:0:0) at 14 days curing period. It was observed that UCS values of all the solidified samples exceeded the landfill disposal limits of 340 kPa, except for the control sample value of 292.1 kPa at 14 days curing period.

Although RHA has been added as a replacement for cement, even at early ages of 7 days, the compressive strength values increases as related to 14 and 28 days. The reasons for early compressive strength development of the mixture with RHA may be due to fineness, amorphous phase, and degree of reactivity of RHA. This increase in strength at early age may partly be due to the pozzolanic reaction, filler effect and to a better distribution of cement particles which produced as a result of adding superplasticizer, as reported by early researches [17-18].
The reduction in strength recorded between the 14 and 28 day of curing can also be credited to the entire consumption of all the mix water in the concrete specimen which stopped the cement hydration process and may be due to the influence of the copper contaminant on the hydration process. This is similar to the results obtained by Safan and Kohoutková [19] in which the samples were exposed to the outside environmental conditions and 15% reduction in strength was recorded between the 7 and 28 days strength.

Table 1. UCS of solidified/stabilised sediment samples throughout 28 days of curing

| Symbols | Mix ratios (S:C:R) | Unconfined Compressive strength (kPa) |
|---------|-------------------|--------------------------------------|
|         |                   | 7 days | 14 days | 28 days |
| A       | 100:0:0           | 346    | 292     | 724     |
| B       | 90:10:0           | 1026   | 1118    | 800     |
| C       | 85:10:5           | 3874   | 1548    | 1295    |
| D       | 80:10:10          | 4326   | 1493    | 881     |
| E       | 75:10:15          | 4522   | 1487    | 1715    |
| F       | 70:10:20          | 1496   | 2092    | 2389    |

S = Sediment, C = Cement, R = Rice husk ash

3.2 Relationship between strength and leachability of copper spiked sediment

The results for the relationship between strength and leachate concentration of copper in the TCLP leaching test at 7, 14 and 28 days is shown in Table 2. As we can observe in the TCLP leachates the maximum leaching concentration of copper were 8.04 mg/l, 7.1 mg/l and 4.73 mg/l while their corresponding strengths were 346 (kPa), 292 (kPa) and 724 (kPa) at 7, 14 and 28 days respectively. The lowest leaching concentrations obtained were 2.28 mg/l, 2.29 mg/l and 0.14 mg/l with each compressive strength of 1496 kPa, 2092 kPa and 800 kPa at 7, 14 and 28 days respectively. From the results discuss above we can see when the compressive strength was high the leachability of copper was reduced.
There is much indication that shows that immobilization is through both sorption processes and by substitution for Ca or Si in the gel phase. Park and Batchelor [20] showed sorption processes are significant in the retention of Cr\textsuperscript{6+}, Cd, Pb, Cu and other metals.

Table 2. Relationship of strength and leachability of Copper in the TCLP at 7, 14 and 28 days

| Mix ratios | UCS 7days (kPa) | TCLP 7days (mg/l) | UCS 14days (kPa) | TCLP 14days (mg/l) | UCS 28days (kPa) | TCLP 28days (mg/l) |
|------------|-----------------|-------------------|-----------------|-------------------|-----------------|-------------------|
| A          | 346             | 8.04              | 292             | 7.1               | 724             | 4.73              |
| B          | 1026            | 3.31              | 1118            | 2.96              | 800             | 0.14              |
| C          | 3874            | 3.10              | 1548            | 2.99              | 1295            | 2.50              |
| D          | 4326            | 2.97              | 1493            | 3.36              | 881             | 2.73              |
| E          | 4522            | 2.64              | 1487            | 2.61              | 1715            | 2.39              |
| F          | 1496            | 2.28              | 2092            | 2.29              | 2389            | 2.28              |

As we can observe in Figure 7 the exponential regression for the unconfined compressive strength with respect to leachability of Copper (Cu) concentrations shows that the leachability of copper is highly related to strength as follows: the compressive strength for the TCLP at 7 days had a Regression value $R^2 = 0.99$ indicating a strong positive correlation of 99% and at 14 days the value of $R^2 = 0.85$ indicating slight decrease of $R^2$ while $R^2 = 0.92$ was obtained at 28 days curing period. All the $R^2$ values obtained indicated a strong relationship between strength and leachability of copper throughout the 28 days period.

![Figure 7. Relation between strength and leachability of copper in the TCLP at 7, 14 and 28 days](image)

4. Conclusions
The unconfined compressive strength (UCS) for the copper spiked sediment after 28 days of curing days was in the range of 292.1 to 4522 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 4522 kPa at 7 days with 15% RHA addition while the lowest strength obtained was 292.1 for the control sample (100% sediment, 0%cement and 0%RHA) at 14 days. All the UCS values
obtained for the solidified sediment samples exceeded the landfill disposal limit of 0.34 Nmm$^2$ (340kPa) for a waste at a disposal site in the UK, except for the control sample at 14 days, which are slightly below the regulatory limit. From the results obtained strength is also related to leachability of the Cu in the TCLP leaching tests at 7, 14 and 28 days curing period. When the strength of the stabilized sediments was low (for the untreated) the leachability was observed to be high, while for the treated specimens with 5-20% RHA addition the leachability decreases with increase RHA accordingly for the stabilized sediment studied.

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