Stabilization of heavy metals in fired clay brick incorporated with wastewater treatment plant sludge: Leaching analysis

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Abstract. Wastewater treatment sludge or known as sewage sludge is regarded as the residue and produced by the sedimentation of the suspended solid during treatment at the wastewater treatment plant. As such, this sludge was gained from the separation process of the liquids and solids. This sludge wastes has becomes national issues in recent years due to the increasing amount caused by population and industrialization growth in Malaysia. This research was conducted to fully utilize the sludge that rich in dangerous heavy metals and at the same time act as low cost alternative materials in brick manufacturing. The investigation includes determination of heavy metal concentration and chemical composition of the sludge, physical and mechanical properties. Wastewater treatment sludge samples were collected from wastewater treatment plant located in Johor, Malaysia. X-Ray Fluorescence was conducted to determine the heavy metals concentration of wastewater treatment sludge. Different percentage of sludges which are 0%, 1%, 5%, 10%, and 20%, has been incorporated into fired clay brick. The leachability of heavy metals in fired clay brick that incorporated with sludge were determined by using Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leachability Procedure (SPLP) that has been analyzed by using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The results show a possibility to stabilize the heavy metals in fired clay brick incorporated with wastewater treatment sludge. 20% of the sludge incorporated into the brick is the most suitable for building materials as it leached less heavy metals concentration and complying with USEPA standard.

1. Introduction
Waste production from wastewater treatment plants especially sewage sludge waste is a major issue towards the environmental nowadays. Massive investigation and research was done during the last 20 years involving wastewater treatment sludge. Several researches have been converting urban sludge into a resource depending on composition and it’s characteristic such as fertilizer in agriculture, industrial chemicals, energy sources, and also material for building [1].

Management of wastewater treatment sludge is one of the most crucial environmental issues in Malaysia due to the rapid increasing in sludge production. With the rapid development growth in the country, amount of wastewater treatment sludge generated increased annually. These wastes needs to be properly managed appropriately and disposed of without causing any harmful environmental effects. According to Azman [2], Malaysia has a production of wastewater treatment sludge generated by municipal and industries sector is 2.97 billion cubic meter per year. Due to the larger amount production of the sludge and increasing population, wastewater treatment sludge consists of various
types of pollutant from domestic and industrial sources. Indah Water Konsortium (IWK) has managed and handed the production of wastewater treatment sludge in Malaysia. Currently, IWK operates and maintains over 4300 public wastewater treatment plants all over Malaysia. Furthermore, the total sludge produce from the entire treatment plants amounts to 5500 metric ton per day or 2.0 million metric ton per year.

Higher amount of wastewater treatment sludge can lead to environmental problem caused by the heavy metals in sludge. The heavy metals in wastewater treatment sludge including chromium (Cr), copper (Cu), nickel (Ni), lead (Pb) and zinc (Zn). Most of the wastewater treatment sludge has been one of the key of environmental problem due to the disposal and sludge handling for instant, landfill and incineration [3,4]. Some researchers have links aluminium contributory influence to occurrence of Alzheimer’s, children mental retardation and common effects of heavy metals accumulation [5]. Furthermore, those are heavy metals which can damage human health and have composition of unhealthy particles [6]. Heavy metals are the principal elements restricting the use of sludge for agricultural purpose [7]. However, disposal of sludge using incineration to ceramic matrices has been proposed but there is some impact towards environment. The process would emit hazardous air pollutants that would cause health effect to people surrounding. Furthermore, most of wastewater and water treatment sludge has been disposed to landfill. In Malaysia, the increasing of wastewater treatment and water sludge in recent years contributed to the higher number of sludge disposed to the landfill. There are 291 landfill sites in April 2007 and about 80% reached their maximum capacity by 2010 and 112 were closed and only 10 sanitary landfills being operate[8].

Thus, recently transforming sludge into raw materials is one the relevant solution overcome environment problem and to achieve good environmental performance. According to some researchers has successfully utilized different types of sludge which obtain provision results [9, 10]. Nevertheless, most of the researches are focusing on physical and mechanical properties include compressive strength, water absorption, density and shrinkage but less attention is towards leachability of heavy metals of the sludge after incorporated into fired clay brick [11,12]. Therefore, in this study the utilization of sludge from wastewater treatment plant was carried to determine the leachability of heavy metals from the sludge towards the environment by using Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP).

2. Materials
There were two sources of wastewater treatment sludge involved in this research namely Type A sludge and Type B sludge located at Johor, Malaysia (figure 1). Upon arriving, the sludge was dried in the oven at 105°C for 24 hours at the RECESS laboratory, UTHM. After the moisture of the sludge was removed, the sludge was grinded and sieve at 200 µm. Next, the clay soil for brick manufacturing was obtained from Yong Peng, Johor and has been kept properly before casting. The soil was dried for 24 hours and grinded in Geotechnical laboratory to eliminate impurities. The material characterization of raw materials were carried out through X-ray fluorescence (XRF) test, performed using Bruker AXS S4 Pioneer.

Figure 1. Type A and type B sludge.
The chemical compositions of the materials are shown in table 1. The results indicated the higher percentage of chemical composition for type A and type B sludge is Silicon Dioxide (SiO\textsubscript{2}) which is 14.30% and 16.30% respectively. Next, the lowest percentage of chemical composition for type A sludge is Barium Oxide (BaO\textsubscript{2}) and and Strontium Oxide (SrO) which is 0.10% respectively. For type B sludge, the lowest percentage is Barium Oxide (BaO) with 0.30%. Meanwhile, for clay soil the result shows that the higher percentage is Silicon Dioxide (SiO\textsubscript{2}) which is 49.30% and followed by Aluminium Sulphide (Al\textsubscript{2}SO\textsubscript{3}) which is 18.4%. Besides that, Titanium Oxide (TiO\textsubscript{2}) shows the lowest percentage which is 0.9% then followed by Magnesium Oxide (MgO) with 0.8%.

| Element                  | Formula   | Concentration (%) | Type A | Type B | Clay Soil |
|--------------------------|-----------|-------------------|--------|--------|-----------|
| Silicon Dioxide          | SiO\textsubscript{2} | 14.3              | 16.3   | 49.3   |           |
| Ferric Oxide             | Fe\textsubscript{2}O\textsubscript{3} | 9.9            | 9.4    | 6.9    |           |
| Phosphorus Pentoxide     | P\textsubscript{2}O\textsubscript{5} | 5.6             | 6.7    |        |           |
| Aluminium Sulphide       | Al\textsubscript{2}SO\textsubscript{3} | 6.7             | 4.8    | 18.4   |           |
| Sulfur Trioxide          | SO\textsubscript{3} | 9.2              | 4.6    |        |           |
| Calcium Oxide            | CaO       | 6.5               | 2.4    |        |           |
| Potassium Oxide          | K\textsubscript{2}O | 0.7             | 1.1    | 3.1    |           |
| Magnesium Oxide          | MgO       | 1.2               | 0.8    | 0.8    |           |
| Titanium Dioxide         | TiO\textsubscript{2} | 0.5            | 0.5    | 0.9    |           |
| Zinc Oxide               | ZnO       | 0.4               | 0.4    |        |           |
| Barium Oxide             | BaO       | 0.1               | 0.3    |        |           |
| Chlorine                 | Cl        | 0.1               |        | -      | -         |
| Strontium Oxide          | SrO       | 0.1               |        | -      | -         |

Meanwhile, abundance of heavy metal elements was founded in raw materials. Only selected parameters were considered based on raw material characterization testing to be tested by using Atomic Absorption Spectrometry (AAS). Henceforth, the analysis of the leachability of fired clay bricks incorporated with wastewater treatment sludge is narrowed down to these few specific heavy metals. The heavy metals selected includes Chromium (Cr), Manganese (Mn), Nickel (Ni), Copper (Cu), Zinc (Zn), Arsenic (As), Cadmium (Cd) and Lead (Pb) as shown in table 2. All concentration values were compared with the concentration limit for heavy metals set by United States Environmental Protection Agency or USEPA (1996) [14].

| Heavy Metals     | Concentration Limit (mg/l) (USEPA, 1996) | Concentration (mg/l) | Type A | Type B | Clay Soil |
|------------------|------------------------------------------|----------------------|--------|--------|-----------|
| Chromium (Cr)    | 20                                       | 0.021                | 0.022  | 0.011  |           |
| Manganese (Mn)   | 260                                      | 3.230                | 1.830  | 0.532  |           |
| Nickel (Ni)      | 8                                        | 0.081                | 0.052  | 0.100  |           |
| Copper (Cu)      | 800                                      | 0.240                | 3.910  | 0.004  |           |
| Zinc (Zn)        | 1200                                     | 7.820                | 5.880  | 0.709  |           |
| Arsenic (As)     | 2.8                                      | 1.040                | 2.560  | 0.000  |           |
The mix proportions of fired clay brick incorporated with wastewater treatment sludge is presented in table 3. In this research, different percentage of sludge incorporated into fired clay brick was manufactured which are 0%, 1%, 5%, 10%, 20% and 30%. However, the 30% of samples were disintegrated during firing thus the testing was conducted up to 20% only based on the properties result. The amount of water and wastewater treatment sludge that used has been stated. The clay, water and sludge was added and mixed together before the sample was pour into brick machine for compaction. The preliminary testing properties was conducted and the result of brick that incorporated with sludge more than 20% show low quality of brick with disintegration effect during firing. Therefore, in this study the maximum mixture of wastewater sludge is 20% in term to produce good quality brick and the leaching testing was conducted.

**Table 3. Mix proportions of fired clay brick incorporated with sludge.**

| Proportion of Type A sludge | Percentage (%) | Weight of sludge (g) | Weight of soil (g) | Percentage of Moisture content (%) | Amount of water (ml) |
|----------------------------|----------------|----------------------|-------------------|-----------------------------------|----------------------|
| 0                          | 0              | 0                    | 2800              | 17.0                              | 476.0                |
| 1                          | 28             | 2772                 |                   | 17.8                              | 498.4                |
| 5                          | 140            | 2660                 |                   | 18.2                              | 509.6                |
| 10                         | 280            | 2520                 |                   | 20.4                              | 571.6                |
| 20                         | 560            | 2240                 |                   | 21.9                              | 613.2                |

| Proportion of Type B sludge | Percentage (%) | Weight of sludge (g) | Weight of soil (g) | Percentage of Moisture content (%) | Amount of water (ml) |
|----------------------------|----------------|----------------------|-------------------|-----------------------------------|----------------------|
| 0                          | 0              | 0                    | 2800              | 17.0                              | 476.0                |
| 1                          | 28             | 2772                 |                   | 17.4                              | 487.2                |
| 5                          | 140            | 2660                 |                   | 18.1                              | 506.8                |
| 10                         | 280            | 2520                 |                   | 20.3                              | 568.4                |
| 20                         | 560            | 2240                 |                   | 21.0                              | 588.0                |

3. Method

3.1. Toxicity Characteristic Leaching Procedure (TCLP)

The method was based on test method 1311 [15] as the USEPA regulatory method. The test procedure was performed to investigate the leachability of heavy metals based on USEPA limit. Firstly, sludges sample was grinded and sieve using 0.7 μm glass fibres. The standard fluid for 1 mol of Sodium Hydroxide (NaOH) and Hydrochloric Acid (HCl) was prepared. Next, the preparation of sample for determining of two extraction fluid was conducted. For extraction number 1, the pH of this fluid was 4.93 then adding glacial acetic acid and NaOH with water. Then, for extraction fluid number 2, the pH of the fluid was 2.88 then adding glacial acetic acid with water without NaOH. After determination of fluid, the sample was prepared in screw-capped polyethylene bottles and the leaching fluid is at ratio 1:20. The bottles were agitated at 30 rpm for 18 hours in an end-over-end manner. The leachate collected was then filtered using 0.7 μm glass fiber filters, preserved the sample before being analyzed using Atomic Absorption Spectrometry to determine the dissolved metals.
3.2. Synthetic Precipitation Leaching Procedure (SPLP)
The method from method 1312 [16] is an agitated extraction that is used to provide information on the mobility (leachability) of organic and inorganic constituents from liquids, soils, and wastes. This procedure is similar to the TCLP but instead of the acetic acid mixture used with the TCLP to simulate landfill leachate, nitric and sulfuric acids are utilized to simulate the acid rain resulting from airborne nitric and sulfuric oxides. If the sample is a waste or wastewater, the extraction fluid employed is the pH 4.2 solution. For this research, extraction fluids with pH 4.2 solution were used. The next procedure is same with TCLP that has been described earlier.

4. Results and discussion

4.1. Leachability of heavy metals by using TCLP
From the results of the TCLP test in Table 4 (Type A) and Table 5 (Type B), it shows that there are insignificant levels of heavy metals leached out from the samples. The concentration of heavy metals for control brick is higher compared to 20% of brick with type A sludge. The concentration of heavy metals has decreased from control to the brick that containing up to 20% of type A sludge. Next, it shows that up to 20% of sludge, the leachability of heavy metals from brick was complying with USEPA concentration limit. Explanations of the results were discussed further according to figure 2a and figure 2b.

Table 4. Leachability of heavy metals concentration of Type A sludge fired clay brick in TCLP.

| Heavy metals | Concentration (mg/l) | Concentration Limit (mg/l) |
|--------------|----------------------|----------------------------|
|              | Control | 1% | 5% | 10% | 20% | USEPA (1996) |
| Chromium (Cr)| 0.027   | 0.031 | 0.033 | 0.033 | 0.033 | 20 |
| Manganese (Mn)| 0.694   | 0.603 | 0.452 | 0.212 | 0.163 | 260 |
| Nickel (Ni)  | 0.009   | 0.004 | 0.005 | 0.006 | 0.008 | 8  |
| Copper (Cu)  | 0.015   | 0.000 | 0.017 | 0.022 | 0.030 | 800 |
| Zinc (Zn)    | 0.418   | 0.503 | 0.482 | 0.418 | 0.403 | 1200 |
| Arsenic (As) | 0.601   | 0.176 | 0.167 | 0.159 | 0.039 | 5  |
| Cadmium (Cd) | 0.000   | 0.000 | 0.000 | 0.000 | 0.000 | 0.8 |
| Lead (Pb)    | 0.011   | 0.012 | 0.012 | 0.010 | 0.009 | 500 |
Table 5. Leachability of heavy metals concentration of Type B sludge fired clay brick in TCLP.

| Heavy metals     | Concentration Limit (mg/l) | Control | 1% | 5% | 10% | 20% |
|------------------|-----------------------------|---------|----|----|-----|-----|
| Chromium (Cr)    | 20                          | 0.027   | 0.012 | 0.014 | 0.014 | 0.038 |
| Manganese (Mn)   | 260                         | 0.694   | 0.345 | 0.292 | 0.291 | 0.163 |
| Nickel (Ni)      | 8                           | 0.009   | 0.004 | 0.004 | 0.004 | 0.026 |
| Copper (Cu)      | 800                         | 0.015   | 0.011 | 0.014 | 0.017 | 0.016 |
| Zinc (Zn)        | 1200                        | 0.418   | 0.433 | 0.396 | 0.341 | 0.311 |
| Arsenic (As)     | 5                           | 0.601   | 0.085 | 0.172 | 0.120 | 0.096 |
| Cadmium (Cd)     | 0.8                         | 0.000   | 0.000 | 0.000 | 0.000 | 0.000 |
| Lead (Pb)        | 500                         | 0.011   | 0.001 | 0.004 | 0.004 | 0.003 |

Figure 2(a). Heavy metals concentration for Type A sludge fired brick using TCLP.

Figure 2(a) illustrated the concentration of heavy metals from control brick and type A sludge incorporated into fired clay brick from TCLP test. In this study, the use of sludge as a partial replacement of clay soil showed positive result towards leachability of heavy metals for type A sludge. The figure shows that the concentration of heavy metals is steadily decreased as the percentage of wastewater treatment sludge increasing. Concentration of heavy metals such as, Manganese (Mn), Zinc (Zn) and Arsenic (As) shown the decreasing concentration of heavy metals compared to control brick. This is also supported by previous researchers that claimed from his study, that all concentration of heavy metals was slightly decreased with the increasing percentages of sludge utilized [5]. However, the trend for Copper (Cu) differ as the value increased with the increasing percentage of sludge. Percentage of heavy metals for control brick is 0.015 mg/l while with added 20% of sludge, the percentage increased to 0.030 mg/l. On the other hand, Cadmium (Cd) obtained very low value of concentration for heavy metals. Concentration of heavy metals for type A sludge up to 20% is still complying with USEPA concentration limit.
Figure 2(b). Heavy metals concentration for Type B sludge fired brick using TCLP.

Figure 2(b) demonstrated the leachability of heavy metals in type B sludge fired clay brick with different percentage of wastewater treatment sludge. Similar to the leachability of type A sludge, the trend has steadily decreased as the percentage of sludge increased. This significant reduction has been expected due to the effect of adding wastewater treatment sludge in the fired clay brick [10]. As for control brick, it shows the highest concentration for Manganese (Mn) then followed by Arsenic (As) and Zinc (Zn) with value 0.694 mg/l, 0.601 mg/l and 0.418 mg/l respectively. Nevertheless, heavy metals such as Chromium (Cr), Nickel (Ni) and Copper (Cu) shows the value less than 0.1 mg/l after additional percentage of wastewater treatment sludge. Nevertheless, all concentration of heavy metals for type B sludge brick is still complying with USEPA standard. This is also agreed by Victoria [11]. as in the study with additional up to 20% of sludge, the leachability of heavy metals for fired clay brick is suitable for construction activities.

On the other hand, table 6 and table 7 summarize the results determined for heavy metals by using the SPLP method for both type A and type B sludge respectively. Further results were then explained according to figure 3(a) and figure 3(b).

Table 6. Leachability of heavy metals concentration of Type A sludge fired clay brick in SPLP.

| Heavy metals | Concentration (mg/l) | Concentration Limit (mg/l) USEPA (1996) |
|--------------|----------------------|----------------------------------------|
|              | Control  | 1%   | 5%   | 10%  | 20%  |                   |
| Chromium (Cr)| 0.002   | 0.004| 0.004| 0.004| 0.027| 20                  |
| Manganese (Mn)| 0.518  | 1.28 | 1.25 | 1.22 | 1.12 | 260                 |
| Nickel (Ni)  | 0.012   | 0.004| 0.005| 0.005| 0.005| 8                   |
| Copper (Cu)  | 0.006   | 0.013| 0.017| 0.015| 0.021| 800                 |
| Zinc (Zn)    | 0.403   | 0.681| 0.637| 0.682| 0.689| 1200                |
| Arsenic (As) | 0.002   | 0.220| 0.378| 0.437| 0.453| 5                   |
| Cadmium (Cd) | 0.000   | 0.000| 0.000| 0.000| 0.000| 0.8                 |
| Lead (Pb)    | 0.000   | 0.004| 0.003| 0.000| 0.000| 500                 |
Table 7. Leachability of heavy metals concentration of Type B sludge fired clay brick in SPLP.

| Heavy metals | Concentration (mg/l) | Concentration Limit (mg/l) |
|--------------|----------------------|---------------------------|
|              | Control | 1%     | 5%    | 10%   | 20%   | USEPA (1996) |
| Chromium (Cr) | 0.002   | 0.004  | 0.005 | 0.004 | 0.003 | 20            |
| Manganese (Mn)| 0.518   | 0.764  | 0.464 | 0.365 | 0.313 | 260           |
| Nickel (Ni)   | 0.012   | 0.006  | 0.008 | 0.010 | 0.015 | 8             |
| Copper (Cu)   | 0.006   | 0.032  | 0.035 | 0.042 | 0.037 | 800           |
| Zinc (Zn)     | 0.403   | 0.395  | 0.333 | 0.287 | 0.251 | 1200          |
| Arsenic (As)  | 0.002   | 0.288  | 0.278 | 0.274 | 0.156 | 5             |
| Cadmium (Cd)  | 0.000   | 0.003  | 0.001 | 0.002 | 0.002 | 0.8           |
| Lead (Pb)     | 0.000   | 0.000  | 0.010 | 0.005 | 0.008 | 500           |

Figure 3(a). Heavy metals concentration for Type A sludge fired brick using SPLP.

Figure 3(a) shows the leachability of type A sludge brick from SPLP test method. Based on the figure, the concentration of heavy metals in type A sludge brick has decreased steadily as the percentage of the sludge is increased. The highest concentration of heavy metals obtained in control brick is Manganese (Mn), and then followed by Zinc (Zn) with value of 0.518 mg/l and 0.403 mg/l respectively. In addition, the concentration of Manganese (Mn) was decreased from 0.518 mg/l for control brick to 0.313 mg/l for 20% of sludge added. This decreasing trend is similar with Zinc (Zn) which is the concentration is decreased from 0.403 mg/l to 0.251 mg/l. The lowest concentration of heavy metals is Copper (Cu), Nickel (Ni) and Lead (Pb). Nevertheless, the heavy metals such as Cadmium (Cd) and Chromium (Cr) obtained very low concentration of heavy metals.
Figure 3(b). Heavy metals concentration for Type B sludge fired brick using SPLP.

Figure 3(b) demonstrated the leachability of type B sludge brick incorporated into fired clay brick. From the figure above, obviously trend was demonstrated that the concentration of heavy metals has decreased steadily as the increasing percentage of sludge. Heavy metals concentration in type B sludge brick has decreased for Manganese (Mn) and increased for Arsenic (As). Nevertheless, it is noted that increasing percentage of sludge has increased the concentration of Arsenic (As) from 0.002 mg/l (control) to 0.453 mg/l with 20% of sludge. Nevertheless, the concentration of Zinc (Zn) has similar trend after additional of 1% to 20% sludge. It is also in accordance to Fytili and Zabaniotou [7] with up to 20% sludge utilization, it is still suitable for construction activities as the concentration of heavy metals do not leach much. Apart from that, all the heavy metals concentration does not exceeding 10 mg/l which means that all the heavy metals comply with the USEPA standard. Furthermore, it is worth to note that result of leaching from both test demonstrated that the quantities of the heavy metals from type B sludge brick containing 20% of sludge are all less than the control brick. It is also supported by Weng et al. [18] that claimed the result is due to the low heavy metals that because most of metals were converted in oxide during the high firing temperature conducted to fire the brick.

5. Conclusion
In terms of leachability test result, this study was investigated the different method of leaching test which are Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP). Both methods has been analysed by using ICPMS. Based on the result, Manganese (Mn) shows the highest value of heavy metals concentration among the other element based on TCLP and SPLP method. Meanwhile, elements include Arsenic (As), Cadmium (Cd), Copper (Cu), Zinc (Zn), Chromium (Cr), and Lead (Pb), has lower concentration for both type of sludge brick. Nevertheless, it shows that type A sludge leach heavy metals lower than type B sludge. In all, due to leachability results from SPLP n TCLP testing, the recommended proportion of sludge in brick is up to 20% as all the heavy metals complied with the USEPA standard. Therefore the proportion is safe to be used as replacement materials in fired clay brick. Long term analysis on leachability could be conducted by using SLT method. Hence, a proper sustainable and safe way of reusing such waste materials as in this research is useful towards the environment and sustainability for construction purposes.
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Acknowledgments
This research was financially supported by Fundamental Research Grant Scheme (FRGS) Vot 1527 under Ministry of Higher Education of Malaysia. Special thanks to Office for Research, Innovation, Commercialization and Consultancy Management (ORICC) from University Tun Hussein Onn Malaysia.