In Situ Remediation of Lead Contaminated Marine Sediment using Bentonite, Kaolin and Sand as Capping Materials

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Abstract. There is a growing public concern over the issue of sediment contamination resulting from industrial and municipal waste discharge, mining activities, and improper use of chemical fertilizer, pesticides, and wastewater irrigation. However, there is a need to reduce the continuous contamination of the water column and biota by the pollutants in the sediment. The conventional approach of contaminated sediment treatment is dredging but is expensive and requires a large area of land for disposal. In situ capping of contaminated sediment is considered as a cheaper technique compared to dredging and efficient treatment technology to immobilize pollutants in sediments on site. A laboratory-scale capping experiment was conducted to evaluate the effectiveness of using bentonite (B), kaolin (K), a mixture of bentonite with kaolin (BK), and sand in immobilizing lead-contaminated sediment using in situ capping treatment technology. The results indicated that applying bentonite, kaolin, and sand as capping materials has the potential to effectively reduce the release of lead from the heavily spiked contaminated marine sediment (3000 ppm) to the overlying water. The adsorption kinetics results obtained showed that the experimental data for adsorption of Pb fitted well to the Elovich, Second-order and Two-constant rate kinetic models as judged by highest values of $R^2$ and the lowest values of standard error of estimate (SE) such as $R^2$ of 0.937, 0.735, 0.932 with SE values of 0.23, 2.62 and 0.26 for B, K and BK respectively in the Elovich, likewise $R^2$ of 0.817, 0.935, 0.821 with SE values 0.33, 0.07 and 0.19 for B, K and BK in the Second-order and $R^2$ of 0.938, 0.637, 0.925 with SE values of 0.000135, 0.0015 and 0.00024 for B, K and BK were obtained for Two-constant rate models, which implied that the process of adsorption was by chemisorption.

1. Introduction
Remediation of contaminated sediments is one of the most difficult and challenging problems in environmental engineering today. One of the primary risks associated with contaminated sediments is bioaccumulation in benthic organisms, which is a route of entry into the food chain. Thus an important goal of sediment remediation is reducing concentrations to these organisms [1]. Presently, due to the regular contamination of the marine sediments by the municipal and industrial waste discharge, improper use of chemical fertilizer and pesticides, wastewater irrigation, and mining activities, public concern has been aroused over the issue [2-4]. Contamination of marine sediments poses a potential threat to marine resources and human health, e.g., through the consumption of seafood. Marine sediments are exposed to a wide range of potential contamination by chemicals that tend to sorb to fine-grained particles. Heavy metals and organic pollutants are the two major kinds of pollutants found in
sediment [5-7]. Moreover, these are toxic contaminants, posing a great risk to the safety of food, ecological environment, and human health. The problem of contamination of marine sediment is widespread in coastal waters in many parts of the world. Most of the heavy metals are hazardous, and they have adverse effects on human health. They may cause serious health problems like upsets on kidneys and brain cells, thereby disturbing their regular function [8]. Further, they can accumulate in the body and result in additional disturbance to health. Lead (Pb) is one of the hazardous elements which is present in industrial effluents. Consumption of trace amounts of lead may result in permanent adverse health effects of brain development.

Generally adopted sediment remediation approaches include dredging, capping of contaminated areas, and monitored natural recovery (MNR). Dredging can remove contaminated sediments, but it requires large areas of land for sediment disposal. It is expensive and may cause secondary contamination of the water column during re-suspension [9-10]. MNR relies on ongoing naturally occurring processes to decrease the bioavailability or toxicity of contaminants in sediment. These processes may include physical, biological, and chemical mechanisms that act together to reduce the environmental risks posed by contaminated sediments. MNR require longer monitoring time and can be even more expensive than for dredging and capping [11]. Capping consists of in situ covering of clean or suitable isolating material over contaminated sediments layer to limit leaching of contaminants, and to minimize their re-suspension and transport. Compared to MNR and dredging, this treatment method is cost-effective, less disruptive, and less time consuming [10, 12]. In situ capping was mostly used for the remediation of polluted sediment and by doing so, improves the quality of the overlying water and the marine habitat [13-14]. This study aims to evaluate the effectiveness of using Bentonite, Kaolin, and sand as capping materials to prevent the release of Pb from the heavily spiked contaminated marine sediment (3000 ppm) to the overlying water.

2. Materials and methods
The dredged marine sediment sample used for this experimental research was collected from the estuary of Sungai Kuala Perlis, Malaysia. It is located between geographical coordinates of latitude 6° 23' 54.88'' N, and longitude 100° 7' 43.55'' E. The sediments collected was later characterized and digested to determine the concentration of inorganic contaminants (heavy metals) according to USEPA Method 3050B. The chemical composition of the sediments, bentonite, and kaolin were determined using an X-ray fluorescence (XRF) spectrometer (Bruker aXS S4 Pioneer, Germany). The sediment sample was spiked with Analytical grade salts of lead (II) nitrate Pb(NO₃)₂ (99%), to obtain an approximately 3000 ppm target concentration to simulate the worst-case scenario of sediment contamination in situ before treatment. The contaminated sediment was allowed to stay for 30 days after spiking to allow homogeneous distribution and adsorption of the contaminants to the sediment soil particle.

2.1. The capping experiment
The concentration level of the heavy metals in the overlying seawater from the contaminated sediment was monitored in a laboratory-scale capping experiment. This experiment was carried out in the wastewater laboratory of the Faculty of Civil and Environmental Engineering, Universiti Tun Hussein Onn Malaysia. This experiment was used to evaluate the effects of capping materials (bentonite, kaolin, and sand) on the release of Pb. For this purpose, four glass tank models (Figure 1) of equal size (35 × 20 × 25 cm) were used, i.e., length 35 cm, width 20 cm, and height 25 cm. About 3.5 kg of contaminated sediment were placed at the bottom of each glass tank model, forming a layer of 5 cm thickness and a 1 cm thick layer (700 g) each of bentonite, kaolin, or sand were placed carefully on the sediment. The seawater was carefully and slowly poured into each model. The total volume of seawater used for each experiment is 7 L. Figure 1 shows the experimental setup; an impeller was installed at the top middle of the glass tanks and was operated at a speed of 100 rpm for 8 hours daily to simulate the ocean wave turbulence to promote the mass transfer of contaminants from the sediment. To investigate the efficiency of using bentonite, kaolin, and sand as capping materials to prevent the release of Pb ions, four different experimental systems were prepared, as shown in Figure 1.
(i) No capping (control)
(ii) 1 cm thickness cap of bentonite + 1 cm thickness cap of sand
(iii) 1 cm thickness cap of kaolin + 1 cm thickness cap of sand
(iv) 1 cm thickness cap of the mixture (50:50) of bentonite + kaolin + 1 cm thickness of sand capping layer above the contaminated sediments.

During the 90 days test period, an aliquot of 20 mL of the overlying water was collected weekly from each glass tanks and tested for lead concentration using AAS.

Figure 1. Sediment capping experimental setup. (a) is the contaminated sediment without any capping (Control), (b) is sediment capped with bentonite and sand (Sed+B+S), while (c) is sediment capped with kaolin and sand (Sed+K+S) and (d) is sediment capped with composite (50:50) of bentonite with kaolin and sand.

2.2. Kinetic study for adsorption of lead

The adsorption kinetics of the Pb ions from sediments was simulated using different kinetic model equations in order to investigate the mechanism of sorption and potential rate-controlling steps such as mass transport and chemical reaction processes, several kinetic models were tested but only three were found to be well fitted by describing the reaction rates of sorption in bentonite (Sed+B+S), kaolin (Sed+K+S) and mixture of bentonite with kaolin (Sed+(B+K)+S) capping materials as judged by the highest values of $R^2$ and the lowest values of standard error of estimate (SE). Thus the three equations model used to simulate the adsorption of pollutants from the sediments were the Elovich equation, the Second-order equation, and the Two-constant rate equations.

The Elovich equation is expressed as

$$ C_o - C_t = a + b \ln t $$

where $a$ and $b$ are constants in each experiment and $C_0$ is the initial concentration of the pollutant in the sediment before treatment while $C_t$ is the concentration of pollutants releasing into the water column at time $t$. It has been widely employed for representing the kinetics of heavy metal ions released and sorbed from sediments or soils [15-16].

While the Second-order equation is expressed as

$$ 1/C_t = 1/C_0 + b_t $$

where $C_t$ is the concentration of pollutants releasing into the water column at time $t$ and $C_0$ is the initial concentration of the pollutant in the sediment before treatment.
The two-constant rate equation has long been used by the chemist to represent the kinetics of chemisorption; thus, it can be applied to simulate the sorption of metals ions by adsorbents. The Two-constant rate equation can be written as follows:

\[ \ln (C_0 - C_t) = a + b \ln t \]  

where \( a \) and \( b \) are rate constants.

\[ SE = \left[ \frac{\sum(C_t - C^*)^2}{n - 2} \right]^{1/2} \]

where \( C_t \) and \( C^* \) are the measured and calculated concentrations of the pollutants sorbed at time \( t \), and \( n \) is the number of measurements.

3. Results and discussion

3.1. Characteristics of the raw materials

The grain size distribution of the sediments was classified into clay, silt, and sand fractions. The sediment in this study was comprised mostly of the silt fraction (81%), followed by clay fraction (16%) and sand (3%). The pH of the sediment is 8.65, which indicates the sample is alkaline, while the natural moisture content is 227.58%. The particle sizes of the capping materials used in this experiment, as shown in Table 1, are as follows: sand (1.38 - 2.00 mm), bentonite (0.10 - 1.18 mm), and kaolin (0.10 - 1.18 mm). The size of the grains is well connected with the surface areas, which have a considerable effect on the adsorption capacity for contaminants. The chemical composition of the sediment is the silica (\( \text{SiO}_2 \)) and Alumina (\( \text{Al}_2\text{O}_3 \)), having 56.89% and 17.26%, respectively, while other components such as \( \text{Fe}_2\text{O}_3 \), \( \text{K}_2\text{O} \), \( \text{Na}_2\text{O} \), \( \text{MgO} \), \( \text{CaO} \), \( \text{TiO}_2 \), \( \text{MnO} \) are also detected in a moderate concentration of less than 10%. The analysis of the sediment indicates moderate pollution of Pb, which is 47% when compared with the EPA sediment quality guidelines. The sediment was then spiked at a high concentration (3000 ppm) before treatment to simulate the worst-case condition of sediment contamination.

The surface areas for sand, bentonite, and kaolin are 0.75, 19.68, and 9.15 \( \text{m}^2\text{g}^{-1} \), respectively. Sand is largely made of more than 75% \( \text{SiO}_2 \), followed by \( \text{Al}_2\text{O}_3 \) and \( \text{CaO} \) in the order of abundance. Similarly, \( \text{SiO}_2 \) (69.75%), \( \text{Al}_2\text{O}_3 \) (15%), and \( \text{Fe}_2\text{O}_3 \) (8.25%) are the main chemical components of bentonite, and kaolin also has an amount of \( \text{SiO}_2 \) (37.62%) and higher amount of \( \text{Al}_2\text{O}_3 \) (27.90%) than other capping material. According to Panda et al. [17], the parent clay mainly contains silica and alumina, which are in major quantities, whereas other oxides such as magnesium oxide, calcium oxide, potassium oxide, zinc oxide, and titanium oxide are present in trace amounts. Other compounds present in this kaolin are potassium oxide (\( \text{K}_2\text{O} \)), magnesium oxide (\( \text{MgO} \)), and several other compounds are present in a small percentage, as shown in Table 1. The cation exchange capacity (CEC) of the bentonite is 73.25 meq/100 g. Generally, bentonite with a high content of montmorillonite (more than 80%) presents CEC values of around 75 meq/100 g [18], while CEC values of the kaolin are 8.52 meq/100 g. The CEC of kaolinite depends on the particle size (both thickness and diameter in the 00I plane and the pH value. The higher pH value gives rise to more negative charges, which lead to a higher CEC value [19].

| Capping material | \( \text{SiO}_2 \) (\%\) | \( \text{Al}_2\text{O}_3 \) (\%\) | \( \text{Fe}_2\text{O}_3 \) (\%\) | \( \text{Na}_2\text{O} \) (\%\) | \( \text{TiO}_2 \) (\%\) | \( \text{CaO} \) (\%\) | \( \text{K}_2\text{O} \) (\%\) | \( \text{MgO} \) (\%\) | Grain size (mm) | Surface area \( (\text{m}^2\text{g}^{-1}) \) |
|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| Sand  | 75.3  | 7.4  | 13.3  | 1.73  | 0.21  | 2.37  | -    | -    | 1.38-2.00  | 0.75  |
| Bentonite | 53.75 | 13.63 | 8.35  | 2.18  | 1.14  | 1.92  | 0.1  | 0.19  | 2.03  | 0.10-1.18  | 19.68 |
| Kaolin | 37.62 | 27.90 | 0.72  | -     | 0.63  | 0.41  | 0.1  | 1.47  | 0.617 | 0.10-1.18  | 9.15  |
| Sediment | 56.89 | 17.26 | 5.36  | 1.92  | 2.14  | 0.83  | 0.1  | 2.18  | 2.33  | -    | -    |
3.2. Release of Pb from the contaminated sediment to the overlying seawater

Table 2 shows the average concentration released of lead (Pb) and pH taken from the overlying seawater throughout 90 days of the experiment as a function of time. Pb can be categorized as trace metals of which a small quantity was released from the capped sediment+bentonite+sand (Sed+B+S) and sediment + (bentonite+kaolin) [Sed+(B+K)+S] and the uncapped (Sed control) sediments. The average concentration release of Pb from the control sediment, sediments capped with bentonite+sand, kaolin+sand, and a mixture of bentonite+kaolin (50:50)+sand to the overlying seawate presented in Table 3. Considering the concentration of Pb measured in the sediment sample after its artificial pollution, its release from the sediment represented a low percentage of the spiked content. The average concentration of Pb measured throughout the 90 days are 6.083 mg/L, 0.771 mg/L, 2.46 mg/L, and 0.847 mg/L for the control, sediment capped with bentonite Sed+B+S, capped with kaolin Sed+K+S and capped with a mixture of bentonite with kaolin Sed+(B+K)+S respectively. This means that higher percentages of the pollutant (Pb) adsorbed onto the sediment remained in the sediment during all the experiment. The EPA sediment quality guidelines give the safe level of Pb as <25 mg/L. The results showed that all the treatments were able to prevent the release of lead to the overlying water efficiently.

Table 2. The average concentration of lead (Pb) releases throughout 90 days experimental period

| Treatment          | Pb concentration (mg/L) | pH   |
|--------------------|-------------------------|------|
| Sed control        | 6.083                   | 6.32 |
| Sed + B + S        | 0.771                   | 9.13 |
| Sed + K + S        | 2.46                    | 7.43 |
| Sed + (B+K) + S    | 0.847                   | 8.97 |

Note: Sed. Control = sediment with no capping
Sed+B+S = sediment + bentonite + sand
Sed+K+S = sediment + kaolin + sand
Sed+(B+K)+S = sediment + (bentonite + kaolin) + sand

The high immobilization rate of Pb by bentonite and mixture of bentonite with kaolin capped sediment was due to the alkaline conditions (Table 2). The adsorption of the cation (Pb) in the clay mineral surfaces is highest at neutral to alkaline pH since negative surface charges dominate, favoring electrostatic attraction of cationic pollutants near the mineral surface. At small pH, protons compete with cations on the adsorption surface sites. High pH reduces the competition due to the predominance of hydroxyl ions (OH-), and more binding sites are available [20-21]. In addition to adsorption, precipitation of Pb hydroxides may also take place at highly alkaline conditions. Bentonite, as a low-cost and effective adsorbent, has significant potential in the application for removing heavy metals from sediments because of its large quantity, high adsorption capability, chemical, and mechanical stability and unique structural properties [22]. Removal of metal ions using bentonite is based on ion exchange and adsorption mechanisms because of the relatively high cation exchange capacity (CEC) and specific surface area of bentonite [22].

3.3. Kinetic study for adsorption of lead (Pb) on the bentonite, kaolin, and bentonite + kaolin adsorbents

The graphs of the adsorption kinetics of lead on the bentonite, kaolin, and bentonite + kaolin using the Elovich, Second-order, and Two-constant rate kinetic models are illustrated in Figures 2 – 4. A plot of Co - Ct against ln t was used to express the experimental data fitted to the Elovich kinetic model, while the plot of 1/Ct against t for second-order and ln (Co – Ct) against ln t for the Two-constant rate kinetic models. All three equations provided a high degree of correlation with the experimental data. The adsorption plots (Figure 2) of Pb by the Elovich model onto bentonite (B), kaolin (K), and bentonite + kaolin (BK). From the analysis of the Elovich model for lead adsorption, a higher R^2 value of 0.937,
0.735, and 0.923 was obtained for the bentonite (B), kaolin (K), and the mixture of bentonite with kaolin (BK) respectively. Equally lower values of standard errors of 0.23, 2.62, and 0.26 for B, K, and BK respectively were obtained. Therefore as judged by the high values of $R^2$ and lowest values of standard error (SE), as can be seen in Table 3, it can be concluded that experimental data fitted well to the Elovich equation and the mechanism of adsorption was through chemisorption.

**Figure 2.** A plot of Elovich equation for Pb sorption on Sed+B+S, Sed+K+S, and Sed+(B+K)+S capping treatments.

**Figure 3.** A plot of Second-order equation for Pb sorption on Sed+B+S, Sed+K+S, and Sed+(B+K)+S capping treatments.

**Figure 4.** A plot of Two – constant rate equation for Pb sorption on Sed+B+S, Sed+K+S and Sed+(B+K)+S capping treatments.
While the $R^2$ values of 0.817 or 0.935 and 0.821 for B, K and BK respectively (Figure 3) were obtained for the second-order model, also with a lower value of the standard error of estimate (SE) = 0.33, 0.07 and 0.19 for the B, K, and BK adsorbents. Similarly, for the Two-constant rate equation (Figure 4) for the lead adsorption, the values obtained for $R^2$ were 0.938, 0.637 and 0.925 for the B, K and BK treatments respectively and lowest values of SE of 0.000135, 0.0015 and 0.00024 for B, K and BK treatments respectively. As we can observe all the three equations used fitted to the experimental data.

The phenomenon of the adsorption process can be attributed to the interaction between the adsorbent and the adsorbate, as well as shows that the nature of adsorption was through a chemical process that happens due to the exchange of valence electrons between the adsorbent and the adsorbate. The adsorption of Pb from the evaluation of the models demonstrated that the mechanism of adsorption of Pb cation was through the process of chemisorption [23]. Table 3 shows the values of $R^2$ and SE obtained for the three equation models for the lead.

Table 3. Correlation coefficient ($R^2$) and standard error of estimate (SE) of the various kinetic equations for Pb sorption

| Equations                          | Sed+B+S| Sed+K+S| Sed+(B+K)+S |
|------------------------------------|--------|--------|-------------|
| Elovich                            |        |        |             |
| $C_0 - C_t = a + b \ln t$          | 0.23   | 0.937  | 2.62        |
| Second–order reaction:             |        |        |             |
| $1/C_t = 1/C_0 + bt$               | 0.33   | 0.817  | 0.07        |
| Two–constant rate:                 |        |        |             |
| $\ln (C_o - C_t) = a + b \ln t$   | 0.000135 | 0.938 | 0.0015      |

4. Conclusion

In this study, an assessment was carried out on the use of bentonite, kaolin, a mixture of bentonite with kaolin and sand as capping materials to prevent the release of lead (Pb) from contaminated marine sediment. The results of this study revealed that bentonite, kaolin, and mixture of bentonite with kaolin effectively reduced the release of lead pollutants in the contaminated marine sediment investigated in this study. Therefore all the capping materials used in this study could be used as potential capping material for preventing the release of Pb from the contaminated marine sediments. The adsorption kinetics of Pb ions from sediments was modelled using three equations, which are the Elovich, second-order, and Two-constant rate kinetic equation models. The experimental data fitted well to these three equations as judged by the highest values of $R^2$ and the lowest values of the standard error of estimate (SE). The adsorption kinetics demonstrated that the studied parameters were adsorbed on the adsorbents by a chemisorption process.

5. References

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