



In Situ Capping Remediation of Contaminated Marine Sediment using Bentonite, Kaolin and Sand to Interrupt the Release of Trace Metals

Mohammed Kabir Aliyu¹, Ahmad Tarmizi Abd. Karim^{1*}, Chee - Ming Chan²

¹Faculty of Civil and Environmental Engineering,
Universiti Tun Hussein Onn Malaysia, Johor, MALAYSIA

²Faculty of Engineering Technology,
Universiti Tun Hussein Onn Malaysia, Johor, MALAYSIA

*Corresponding Author

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Abstract: In situ capping remediation is a technique by which a capping material is placed on top of sediments to prevent continued contamination of surface water from the contaminated sediments. In this study Bentonite, Kaolin, Sand and mixture of Bentonite with Kaolin were used as capping materials. The objective of this study was to investigate the possibility of using Bentonite, kaolin, mixture of Bentonite with kaolin (1:1) and sand as capping materials to block the release of trace metals (Pb, Cd, Cr, Cu, and Zn) from the artificially contaminated marine sediments, A bench scale laboratory experiment in aquariums was performed for a period of 90 days where 1-cm-thick layer of capping materials was placed above the contaminated sediments. The average Pb concentration in control, capped with bentonite, kaolin, and mixture of both is 4.08, 0.77, 6.77 and 0.84 mg/l respectively, similarly for Cr is 5.63, 0.15, 5.63 and 0.29 mg/l respectively. The average concentration for Cu is 4.28, 0.145, 5.63 and 0.11 mg/l respectively and also for Zn and Cd which are the most mobile is 68.81, 5.82, 75.56 and 23.46 mg/l and 58.35, 14.75, 59.97 and 39.8 mg/l respectively. The trace metals were released into the overlying water from the heavily contaminated sediments in the following decreasing order Cd > Zn > Cu > Pb > Cr. For the uncapped all the contaminants are constantly released with various kinetic depending of mineral oxidation or dissolution or leaching. The release did not go beyond 32 % of the initial pollutants in the sediments. Bentonite and its mixture with Kaolin have much higher metal adsorption capacity compared to that of only Kaolin. The results showed that the capping materials Bentonite and mixture of Bentonite with Kaolin were effective in interrupting the release of Pb, Cu and Cr throughout the experimental period but Cd and Zn were only captured from the beginning but later released gradually until completion. It was concluded that Bentonite and its mixture additive can be used as potential capping materials for the remediation of contaminated sites.

Keywords: In-situ capping, heavy metals, contaminated sediment, bentonite, kaolin, sand

1. Introduction

In situ capping of contaminated marine sediments is a remediation technique in which a capping material is placed on top of contaminated sediments so as to prevent the incessant contamination of surface water and biota above the contaminated sediments. Generally accepted sediment remediation approaches currently include dredging, monitored natural recovery (MNR) and capping of contaminated areas. Dredging can remove contaminated sediments but it

*Corresponding author: tarmizi@uthm.edu.my

requires large areas of land for the disposal of sediment. It is expensive and may cause secondary contamination of the water column during re-suspension (Francingues et al., 2008 & Gosh et al., 2011). MNR depend 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 environmental risks posed by contaminated sediments. MNR require longer time monitoring and can be even more expensive than for dredging and capping (Magar et al., 2009). Capping consist 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 cheaper, less disruptive and less time consuming (Palermo, 1998).

Capping can be divided into two categories, which are passive or active capping depending on the material used for the capping. In passive capping, an inert material such as sand, gravel, or other non-reactive materials are usually used to isolate the contaminated sediments, to lower contaminant migration, and to discourage burrowing and bioturbation for the benthic communities (Hull, Jersak & kasper, 1999). Passive capping does not effectively interrupt the release of toxic contaminants, and it requires a high thickness of passive caps to isolate contaminants in marine sediments (Knox et al., 2008). Compared with passive capping, active or reactive capping uses the materials that react with the sediment contaminant, block the release of the toxic compounds from the sediments, and reduce the bioavailability of the contaminant.

The research on the capping materials for the control of contaminants in the sediments has been conducted and different capping materials were used for each contaminant. While organic contaminants can be bio-degraded by microorganisms, heavy metals are non-biodegradable there by accumulates in the sediments. Different capping materials to bind and block heavy metals from the sediments were investigated. Heavy metals have been reported to be effectively immobilized by including apatite, red mud, calcium phytate and mineral rock phosphate (Knox, Kaplan & Paller, 2006; Basta & McGowen, 2004). Other natural minerals such as zeolite and calcium minerals were also used for capping material to block the release of heavy metals (Kang, Gu & Park, 2016; Jacobs & Förstner, 1999).

In this study the applicability of bentonite, kaolin and mixture of bentonite with kaolin which to the best of our knowledge, have not been used as an active capping material for remediating contaminated marine sediments by other researchers was investigated.. Bentonite and kaolin can easily be obtained due to their low cost and high availability, sand was also used in the experiment as a capping material to prevent the bentonite and kaolin from floating and give them more support against erosion. The evaluation was based on the ability of the capping materials to block the release of toxic metals (Pb, Cd, Cr, Cu, and Zn) from the marine sediments and to stabilize these toxic metals in the sediments. Although most of previous researches were done in batch experimental methods and few studies were done in a flow condition, all of the experiments here were performed under a dynamic condition using an aquarium. An impeller was also attached to the aquarium to simulate ocean wave turbulence.

2. Material and Method

The capping materials used are bentonite, kaolin and sand and their physical and chemical characteristics were analysed. bentonite was provided by High Tech Marketing Company (Malaysia), The kaolin used was obtained from kaolin Malaysia Sdn. Bhd and the sand was obtained from a local supplier in Batu Pahat. All capping materials were used without any purification. A specific surface area was determined using the nitrogen adsorption isotherm technique at 77 K using an Autosorb-iQ surface area analyzer (Quantachrome Corp., USA). The data obtained from the surface area analyzer were interpreted using the Brunauer-Emmett-Teller (BET) method. X-ray fluorescence (XRF) analysis was performed using an XRF spectrometer (Bruker aXS S4 Pioneer, Germany) to quantify the elemental composition of bentonite, kaolin and sand.

3. Sediment Collection and Spiking

The sediment was collected using the backhoe dredger from the seashore of Kuala Perlis. The site is located between geographical coordinates of latitude 6° 23' 54.88" N and longitude 100° 7' 43.55" E. It was later stored in the Laboratory at 4°C after collection for further experiment. The samples were then dried at ambient temperature, and any debris was removed by sieving with a 1mm sieve. The sediments collected was then characterised and the concentration of inorganic contaminants (heavy metals) was determine qualitatively to know the general concentration of all the elements and quantitatively for the selected metals using inductively coupled plasma-mass spectroscopy (ICP-MS) ELAN 9000, Perkin Elmer, USA) after acid digestion according to Edgell (1989) (USEPA Method 3050B) of the samples.

2.3 Sediment Contamination by Spiking

Preliminary analysis of the original sediment sample indicated that the sediment contains low concentration of the toxic heavy metals. ICP-MS was used because it can detect heavy metals contamination even at low concentration. Therefore the collected sediment sample was artificially polluted. To artificially contaminate the sediment sample,

analytical grade salts of Lead nitrate $Pb(NO_3)_2$ (99%), Copper (II) nitrate trihydrate $Cu(NO_3)_2 \cdot 3H_2O$ (99%), Cadmium nitrate tetra hydrate $Cd(NO_3)_2 \cdot 4H_2O$ (99%), Chromium (III) nitrate nona hydrate $Cr(NO_3)_3 \cdot 9H_2O$ (99%) and Zinc chloride ($ZnCl_2$) (99%) were dissolved in deionized water prior to addition into the sediment. The sediment/slurry was then mechanically mixed daily to ensure homogeneous distribution of the contaminants in the sediment. The sediment sample was spiked with the above selected heavy metals to obtain an approximately 3000 ppm target concentration so as to simulate worst case scenario of sediment contamination in situ before treatment. The contaminated sediment was allowed to stay for a period of 30 days after spiking so as to allow homogeneous distribution and adsorption of the contaminants to the sediments soil particle.

2.4 The Capping Experiment Set Up

The Bench-scale capping experiments was conducted in the waste water laboratory of the Faculty of Civil and Environmental Engineering, Universiti Tun Hussein Onn Malaysia. For the purpose of the experiment four glass aquariums (Figure 1) of equal size ($35 \times 20 \times 25$ cm) were set up. Each aquarium contains 3.5 kg of contaminated sediment at the bottom forming a layer of 5cm thickness and a 1cm thick layer (700g) each of bentonite, kaolin or sand on the sediment. The top part holds the sea water. The total volume of seawater used for each experiment is 7 Litres. An impeller was attached on the top at the middle of the glass aquariums as shown in Figure 1, this is to replicate ocean wave and was operated at a speed of 100 rpm for 8 hours daily to maintain water turbulence and promote mass transfer of contaminants from the sediment, the speed is equivalent to 3.4 Km/hr in the field. Four experimental setups were designed to investigate the efficiency of each capping material: (i) No capping i.e. control sediment (ii) Sediment capped with 1cm thickness each of bentonite and sand (iii) Sediment capped with 1cm thickness each of kaolin and sand (iv) Sediment capped with 1cm thickness each of the mixture (50:50) of bentonite/ kaolin and Sand. Seven Litres of clean synthetic seawater was carefully poured into each of the four aquariums. Over the 90 days test period, an aliquot of 20mL of the overlying water was collected randomly from different sections of the aquarium at every second day of the start of the test and then weekly from each aquarium using a plastic syringe connected to plastic tube. The pH and electrical conductivity (EC) of seawater was measured using pH/EC meter (HQ 440D multi, Hatch, USA), and the concentration of each element (Pb, Cd, Zn, Cr, Cu) in the overlying water were monitored during the whole duration of the experiment (90 days). The samples taken was filtered with a $0.45 \mu m$ membrane filter and acidified with 0.2% concentrated HNO_3 . The concentrations of the heavy metals in the samples were determined using inductively coupled plasma optical emission spectroscopy (ICP-OES, Optima 8000, Perkin-Elmer, USA). The schematic diagrams as shown in Figure 2 shows the nature and depth of the capped (A) and the uncapped sediment (B) with the capping layers arrangement.

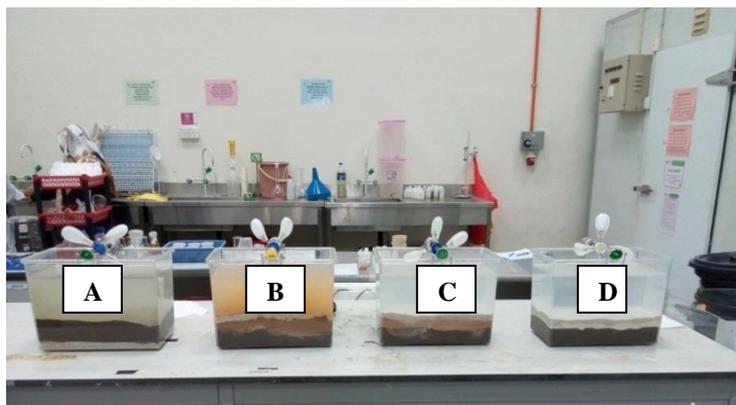


Fig. 1 - Sediment capping set up in aquariums: A is the control sediment, B is sediment capped with bentonite and sand, while C is sediment capped with kaolin and sand and D is sediment capped with mixture of bentonite with kaolin (1:1) and sand

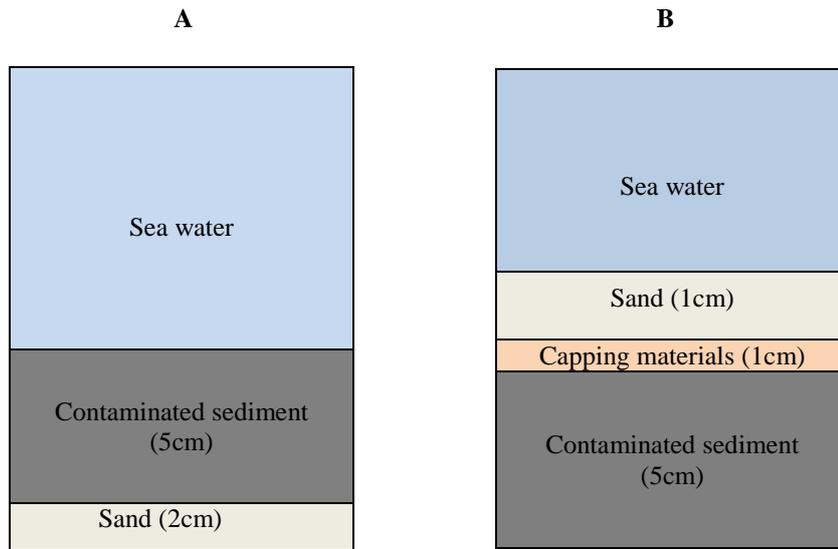


Fig. 2 - The schematic diagrams of experimental settings about the nature and depth of capping materials for (A) uncapped sediment and (B) capped contaminated sediments

3. Results and Discussions

3.1 Characteristics of the Raw Materials

Table 1 presents the physicochemical characteristics and background concentration of heavy metals in the marine sediment while Table 2 presents the physical and chemical properties of the raw materials for the capping experiment. The sediment in this study was composed mainly of the silt fraction, followed by clay fraction and sand. The pH of the sediment is 8.65 which indicate alkalinity pH value of the sediment while the average moisture content was 227%. The grain sizes of the capping materials used in this study 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 highly connected with the surface areas, which have a significant effect on the adsorption capacity for contaminants. The surface areas for sand, bentonite and kaolin are 0.75, 19.68, and 14.72 m² g⁻¹, respectively. Sand is mainly composed of more than 75% SiO₂, followed by Al₂O₃ and CaO in the order of abundance. Similarly SiO₂ (69.75 %), Al₂O₃ (15.00%) and Fe₂O₃ (15.60%) are the main chemical components of bentonite, and kaolin also has amount of SiO₂ (37.62%) and higher amount of Al₂O₃ (27.90) than other capping material.

The concentrations of metals in the sediment before spiking were compared with sediment quality guidelines proposed by EPA (1997). The contaminants concentrations according to the sediments guide lines indicates low concentration of the toxic metals (Pb,Cr,Cu, Zn and Cd) in the marine sediments sampled and may not cause harm to the benthic organism and humans as well. This is the reason why the sediment was spiked at 3000 ppm to simulate worst case scenario of sediment pollution. Assessing environmental and geochemical condition of metals in sediment by comparison with Sediment quality guideline shows that Pb, Cr and Cd are slightly polluted, while Cu and Zn are non-polluted in this study area.

Table 1 - Characteristics and background concentration of metals in the dredged marine sediment

Trace metals content before spiking (mg/kg)					Soil texture (%)			pH
Pb	Cr	Cd	Cu	Zn	sand	silt	clay	
51	28	1.69	14	71	7	78	15	8.65

Table 2 - Chemical composition and physical properties of Sand, bentonite and kaolin used as capping materials

Capping materials	Chemical composition (%)									Physical properties	
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	Na ₂ O	TiO ₂	CaO	C	K ₂ O	MgO	Grain size (mm)	Surface area(m ² g ⁻¹)
Sand	75.3	7.4	1.3	1.73	0.21	2.37	-			1.38-2.00	0.75
Bentonite	69.75	15.00	15.60	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	14.36

3.2 The release of Pb, Cr, Cu, Cd, Zn from Contaminated sediment to the overlying sea water.

The release trend of the 5 heavy metals (Pb, Cu, Cr, Cd and Zn) in the capped and uncapped contaminated sediments for the period of 90 days is shown in Figure 4, 5, 6 and 7. Pb, Cu and Cr can be classified as trace metals of which small amount was released from the capped and uncapped sediments to the overlying sea water throughout the 90 days period of the experiment. As we can observe in the control sample only Cd and Zn were released at higher concentration but in the bentonite + sand capping treatment Pb, Cr and Cu were more suppressed then followed by Zn and Cd (Figure 4) all through the 90 days experiment. For the kaolin + Sand treatment (figure 5) the trace metals were released into the overlying water from contaminated sediments in the following decreasing order: Cd > Zn > Cu > Pb > Cr. While for the (bentonite+kaolin) + Sand treatment (figure 6) was also able to interrupt the release of the elements (Pb, Cr and Cu) significantly. The release of Cd and Zn lasted for a longer period more than Pb, Cu and Cr. Cd was the highest release then followed by Zn in the five trace metals investigated in this study and their release was prolonged until the completion of the 90 days experiment. The high mobility of Cd is in accordance with the findings of several other researchers. Stephens et al. (2001) reported that Cd and Zn were more mobile than Pb, Cu, Ni, Zn, and Fe. Electronegativity, electrostatic force, or metal hydrolysis significantly influenced the mobility of trace metals in the sediments (McBride, 1994). The high mobility of Cd and Zn can be attributed to their low hydrolysis reaction equilibrium constant, as compared with other trace metals

Figure 4 and Table 3 shows that the release of Cd, the most mobile metal, varied significantly depending on the capping material. The Cd concentrations in the seawater overlying uncapped sediment and those capped with bentonite, kaolin and mixture of bentonite with kaolin are 58.35, 14.75, 59.97 and 39.8 mg/l respectively. This indicates that among the capping materials used in this study, bentonite capping is the most effective method for interrupting Cd release. The average Pb concentration in control, capped with bentonite, kaolin, and mixture of both is 4.08, 0.77, 6.77 and 0.84mg/l respectively, similarly for Cr is 5.63, 0.15, 5.63 and 0.29 mg/l respectively. The average concentration for Cu is 4.28, 0.145, 5.63 and 0.11mg/l respectively and also for Zn which also one of the most mobile is 68.81, 5.82, 75.56 and 23.46 mg/l respectively. From this result we can observed that bentonite and mixture of bentonite with kaolin effectively blocked the release of Pb, Cr and Cu from the sediments to the overlying seawater more than the kaolin alone capping treatment.

More on this Singh et al. (1998) reported the order of metal mobility as Cd = Zn > Ni > Cu > Pb for dredged sediment. Both our experimental data and those of previous studies have shown that Cd and Zn were more mobile and less adsorbed to the sediment surfaces than other trace metals, heavy metals including Cd, Cu, Ni, Pb, and Zn have different mechanisms associated with the adsorption to solid surfaces. The adsorption of cationic metal ions on the surface of solids is strongly influenced by the charge-to-radius ratio, metal electronegativity, and the hydrolysis constant (Kang, Lee & Choi, 2016). Blocking efficiency 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 (Shi et al., 2011).

3.3 Changes in pH of the Overlying Sea Water

The differences in pH in the seawater overlying the capped and uncapped sediments during the experimental run are shown in figure 4.5; this difference is identified for significantly influencing the behavior of trace metals. The average pH levels throughout 90 days period of the bentonite, kaolin and mixture of bentonite with kaolin treatment were 8.92 ± 0.16, 6.49 ± 0.11 and 8.58 ± 0.13 respectively, which is higher than that in the uncapped condition which is 6.86 ± 0.12. However, the average pH of the kaolin treatment was 6.49 ± 0.11 which is almost similar and a bit lower to that of the control condition. On the other hand the pronounced alkalinity of Na bentonite capping treatment is related to

increase in pH with the addition of bentonite and which can at least partially be attributed to the hydrolysis of montmorillonite (Na^+ is exchanged for H^+ of water) as suggested by Kamil & Shainberg (1968) and Bar-on & Shainberg (1970). The Na^+ is exchanged for H^+ which leaves OH^- in solution, thereby increasing the pH. Higher pH levels can be expected in seawater overlying Bentonite and mixture of bentonite and kaolin capped sediments than that in the uncapped and kaolin capped conditions. This higher pH leads to lower mobility of the cationic trace metals by increasing their adsorption in the sediments capped with bentonite and mixture of bentonite with kaolin treatments. However, their differences were not that significant. Increasing pH has implications upon the magnitude of electric charges, i.e., increases the negative charges in surface clay and subsequently increases cationic heavy metal adsorption. The adsorption of cations (Cd, and Zn) on mineral surfaces is maximum at neutral to alkaline pH because negative surface charges predominate, favoring electrostatic attraction of cationic pollutants towards the mineral surface. At low pH, protons compete with cations on the adsorption surface sites. High pH decreases the competition due to prevalence of hydroxyl ions (OH^-) and more binding sites are available (Duan & Su, 2014; Huifen et al., 2011). In addition to adsorption, precipitation of Cd and Zn hydroxides may also occur at highly alkaline pH.

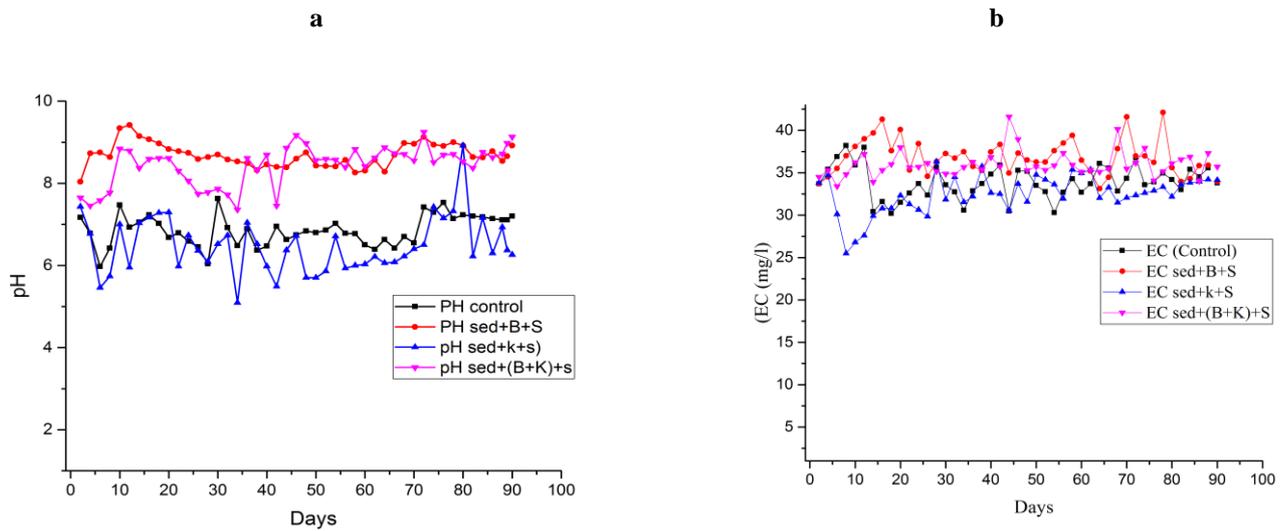


Fig 3 - (a) pH and (b) EC of the seawater above the uncapped sediments and those capped with bentonite, kaolin and mixture of bentonite + kaolin (1:1) respectively thought out 90 days period

Table 3 - Average trace metal concentrations in the overlying seawater for the uncapped and sediments capped with bentonite, kaolin and mixture of bentonite with kaolin.

Contaminants	Sed. Control (mg/l)	Sed+B+S (mg/l)	Sed+K+S (mg/l)	Sed+(B+K)+S (mg/l)
Pb	4.08	0.771	6.77	0.847
Cr	5.63	0.158	5.63	0.293
Cu	4.28	0.145	5.67	0.118
Cd	58.35	14.758	59.97	39.8
Zn	68.81	5.82	75.56	23.46

Note: Sed. = sediment
 B = bentonite
 K = kaolin
 S = sand

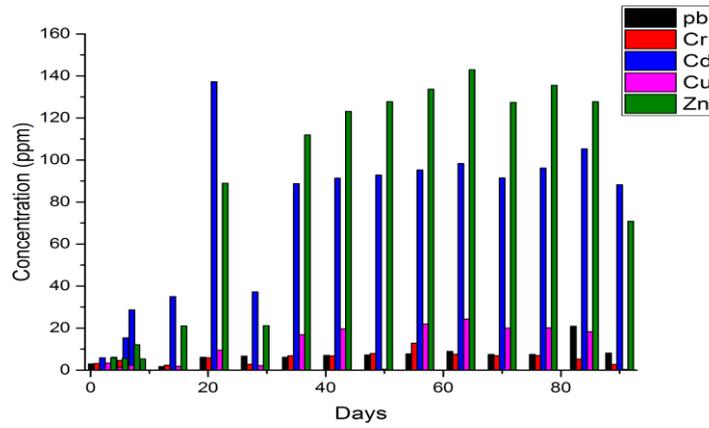


Fig. 4 - Release of Pb, Cu, Cr, Cd and Zn from control sample for 90 days period

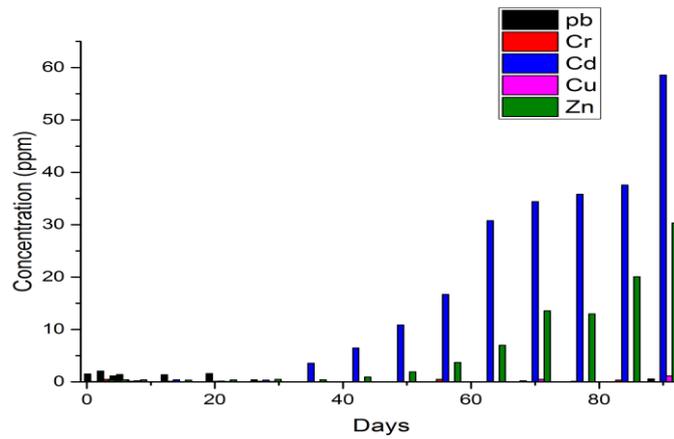


Fig. 5 - Release of Pb, Cu, Cr, Cd and Zn from bentonite + Sand sample for 90 days period

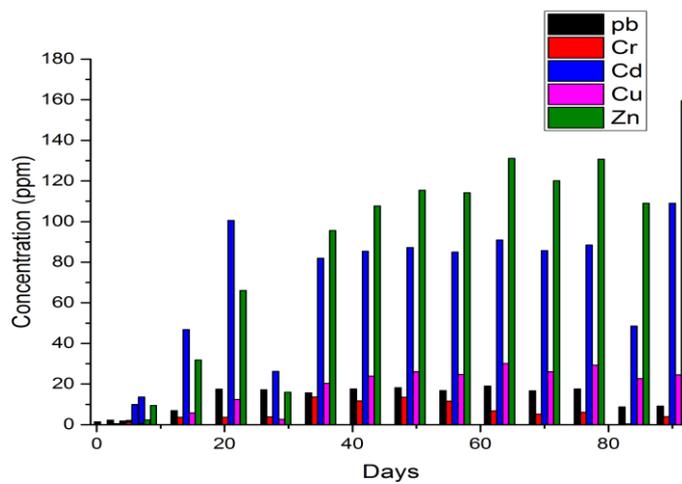


Fig. 6 - Release of Pb, Cu, Cr, Cd and Zn from kaolin + Sand sample for 90 days period

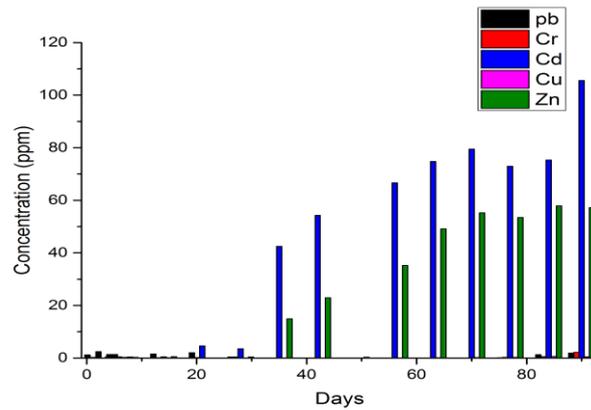


Fig. 7 - Release of Pb, Cu, Cr, Cd and Zn from (bentonite + Kaolin) +Sand capping layer for 90 days period

4. Conclusions

In this study, an assessment was carried out on the use of Bentonite, Kaolin, mixture of bentonite with kaolin and sand as capping materials to reduce the release of trace metals from marine sediments and to sequester those pollutants in contaminated marine sediments. The sediment sample was artificially polluted with Pb, Cr, Cu, Cd and Zn. Then it was capped with bentonite, kaolin and mixture of both (50:50) to test the capture efficiency of the clay minerals. It was observed higher pH leads to lower mobility of the cationic trace metals by increasing their adsorption in the sediments capped with Bentonite and the mixture of Bentonite with kaolin treatments. The results of this study revealed that bentonite and mixture of bentonite with kaolin significantly reduced the release of pollutants investigated in this study. However, all capping materials used in this study could be used as a potential capping material for interrupting the release of Cd, Cu, Ni, Pb and Zn from the contaminated marine sediments.

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