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Use of Bentonite and Zeolite to Stabilize Nutrients and Heavy Metals Within Earthen Pond Sediments

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ABSTRACT



Leaving out the sediments of earthen ponds for several years without disinfection or treatment leads to high concentrations of nitrogen and phosphate compounds, heavy metals and increasing sediment pollutants which release into the water. Therefore, this study aimed to evaluate the effectiveness of capping or mixing of bentonite and zeolite in different percentages for the stabilizing of nutrients and heavy metals in earthen pond sediments for interrupt their release to the water. The pH and electrical conductivity (EC), NH₄-N, NO₃-N, PO₄-P and heavy metals (Cd, Cu, Fe, Mn, Ni, Pb and Zn) in the sediments were measured by 45-day laboratory incubation experiments. The results showed that the highest stabilization efficiency of NH₄-N, NO₃-N, Cd, Cu, Pb and Zn in the sediments was recorded with zeolite capping of 6% of the sediment weight (ZC 6%) by 65.4%, 41.8%, 66.0 %, 63.9 %, 54.2 % and 57.3 %, respectively, while, the highest stabilization efficiency of PO₄-P, Fe, Mn and Ni was with bentonite capping of 6% of the sediment weight (BC 6%) by 61.9%, 42.7 %, 57.0 % and 52.6 %, respectively. Therefore, it is recommended to use bentonite or zeolite as capping material for sediment at a rate of 6% at the bottom of the pond can be effective for the control of nutrients and heavy metals when they are increased in earthen pond sediments for interrupt their release into water.

keywords: Bentonite - Zeolite - Nutrients - Heavy Metals - Sediments - Earthen Pond.

INTRODUCTION

In recent years, fast development has occurred in all forms of aquaculture activity, including the development of hatcheries and feed mills. This resulted in increases in farm productivity and profitability. However, there is much concern about ecosystem health and food security (Abdel-Meguid *et al.*, 2005; Ali *et al.*, 2006). Previous studies of intensive fish farming have shown that some toxic substances present in some feed ingredients, fertilizers added to ponds (urea, phosphate and poultry manure), residues with fish treatments, uneaten feed remains, faecal wastes from fishes may have some negative effects which represent the main cause of pollutants in water and sediment (Ali *et al.*, 2006; and El-Kholy, 2013). Among these pollutants, the high proportion of nutrients from nitrogen and phosphate compounds that enhancing the process of eutrophication which is most prevalent water quality problem (Bhagowati and Ahamad, 2019; Lee *et al.*, 2019). Also, the high concentration of heavy metals may adversely affect the sediments ecosystem safety, water quality and fish production, and also the human health (Adriano 2001; Zhou *et al.*, 2004). The high nitrogen, phosphate and heavy metals concentration can cause suitable environment for the spread of many fish diseases leading to fish mortality (Ali and Abdin, 2003; Tohamy *et al.*, 2006).

Sediments occur in aquatic ecosystems in two major compartments, through depositional processes and when suspended in solution, which deposited as a layer of particles on the bottom of water bodies (Davies and Abowei, 2009). Sediments are normally mixtures of several components such as different minerals and organics debris (Habes and Nigem, 2006). Sediments is possible source for

water pollution and responsible of releasing the nutrients into the water column (Osman and Kloas, 2010; Yin *et al.*, 2016). Pollutant metals are often weakly bound within sediments, facilitating their chemically or biologically mediated release into waters (Blasco, *et al.*, 2000). In natural aquatic systems, the geochemical processes responsible for the exchange of metals at the water-sediment interface are adsorption, precipitation, ion exchange and polymerization (Ayari *et al.*, 2005). These processes are principal mechanisms to convert soluble to more stable solid phases by natural occurring or artificial additives such as lime material, phosphate, zeolite and bentonite (Chen *et al.*, 2000; Cao *et al.*, 2003; Ciccu *et al.*, 2003, Peggy and Vlassopoulos, 2010). Adsorption is one of the simplest and most cost-effective techniques compared to membrane, ion exchange and electrochemical processes (Vereš and Orolínová, 2009; Varedaa, *et al.*, 2019). Such mechanisms depends on a number of external environmental factors such as pH, ionic strength, the type and concentration of organic and inorganic ligands and the available surface area for adsorption caused by the variation in grain size distribution, anthropogenic input (Awofolu *et al.*, 2005). Kabata-Pendias and Pendias (1992) reported that solubility of most metal ions decreases with increasing soil pH.

Bentonite is absorbent swelling clay consisting mostly of montmorillonite. The montmorillonite is an aluminum phyllosilicate mineral with a very large total surface area, which makes bentonite a highly adsorbent, with high cation exchange capacity. The ion exchange process in bentonites is influenced by several factors such as concentration and nature of cations and anions, pH value and crystal structure of the bentonite (Vereš and Orolínová, 2009). Bentonite is

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considered as low-cost and applicable for heavy metals sorption from polluted sediment and water (Zuzana and Michaela, 2015; Hussain and Ali, 2021). Zeolite is hydrous aluminosilicate minerals that occur naturally but can also be synthesized (Anis *et al.*, 2016). Aluminosilicate is connected with properties of zeolites, including high adsorption capacity, with relatively low cost and ecological compatibility (Akimkhan, 2012). The natural zeolites contained a complement of exchangeable Na, K and Ca ions that treatment improved the removal efficiency for metals through ion-exchange processes (Curkovic, *et al.*, 1997).

In some previous studies (Ali, 2014; Miltiadis *et al.*, 2015; Jia *et al.*, 2016; Chunhui *et al.*, 2018; Gu *et al.*, 2019; Alvarado *et al.*, 2020) the methods for sediments contaminated remediation with NH₄-N, NO₃-N, PO₄-P and heavy metals using bentonite and zeolite in lakes, fish farms and agricultural fields were differed in order to stabilize them and not release from sediments or soil to the water or to crops. Most of these studies were conducted in the ex situ laboratory.

The study aimed to evaluate the effectiveness of capping or mixing of bentonite and zeolite in different percentages for the stabilizing of nutrients and heavy metals in earthen pond sediments for interrupt their release to the water. Thus, decreases the rate of water change and avoid the spread of many diseases that can cause the fish mortalities and low productivity.

MATERIALS AND METHODS

Study area

The present study was conducted in Delta Barrage Research Station (DBRS) which belongs to Channel Maintenance Research Institute (CMRI) at the National Water Research Center (NWRC) in Egypt. The station contains a number of rearing earthen ponds for grass carp fingerling that receive their water from the Nile River. As the fingerlings rearing period and before transferring the fry produced from the artificial breeding of fish to the rearing ponds, the ponds are fertilized with urea, phosphate and poultry manure to produce phytoplankton and zooplankton to the fry feeding at the beginning. After about a month, when the fry gained more weight, they fed on artificial pellet at 25% protein until the end of the fingerlings rearing period. After the end of rearing season, surface sediment was collected from about 100 - 150 mm from the ponds floor from 5 ponds

(P1-P5) using sediment sampler to performed chemical measurements and incubation experiments on sediment.

Incubation experiments and extraction amendments from sediment

Surface sediment was collected from about 100 - 150 mm from the ponds floor (from pond P1 to pond P5) using sediment sampler. Sediment samples from the five ponds were air-dried, crushed, passed through a 2-mm mesh screen and stored in polyethylene bags for analysis. The physo-chemical properties of sediment samples were measured to select the sediments most loaded with nutrients and heavy elements among the five ponds.

Then, Laboratory experiments using different treatments of bentonite and zeolite were performed on the sediments of the pond that most loaded with nutrients and heavy metals, to determine their ability to stabilize nutrients and heavy metals into pond sediments for interrupt their release to the water. A numbers of samples were taken from the dried sediments. Each sample weighed 200 g and packed in 250 g plastic pot. Grain sizes of bentonite and zeolite were supplied of 20 - 60 µm, 1.2–2.4 mm, respectively, and used without cleaning. The chemical composition of bentonite used is Al₂H₂Na₂O₁₃Si₄, while for zeolite is NaAlSi₂O₆-H₂O. Incubation treatments of sediment by bentonite and zeolite were performed at rates of 4 % and 6 % of the sediment weight (i.e., 4 % or 6% weight of metal/100% weight of sediment) for each of them, as following: Nine setups were prepared, as shown in Fig. 1: one is control (Ctrl), two treatments for sediment mixed with bentonite 4 % (BM 4%) and bentonite 6 % (BM 6%), two treatments for sediment capped with the same percentages of bentonite 4 % (BC 4%) and bentonite 6 % (BC 6%), two treatments for sediment mixed with zeolite 4 % (ZM 4%) and zeolite 6 % of zeolite (ZM 6%), two treatments for sediment capped with the same percentages of zeolite 4 % (ZC 4%) and zeolite 6 % of zeolite (ZC 6%). Each treatment was repeated five times for different incubation periods i.e. (3 periods of time) 15, 30 and 45 day (Total samples = 9 treatment × 5 replicates × 3 times = 135 samples). The pots were wetted to field capacity (field capacity = 21 %) by the addition of distilled water, and incubated at room temperature (25°C ± 2). Distilled water was added daily to maintain the moisture content at the field capacity. The sediment was removed from the pots at each specific time period for performing chemical analyzes.

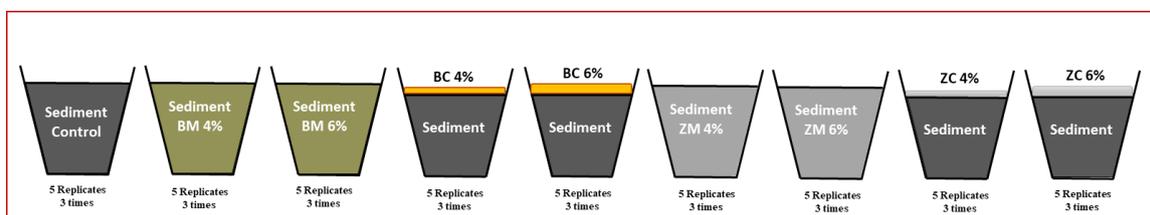


Fig. 1. Schematic diagram of incubation experiments for sediment remediation

Chemical analyses of sediments

pH were measured at 1:2.5 sediments to water ratio suspension (Thomas, 1996), while electrical conductivity were measured at 1:5 sediments to water ratio extracts as soon as the samples reached the laboratory (Jakson, 1973). For the determination of heavy elements, 2 g of each sediments sample was digested with 15 ml of aqua-regia (1: 3 HCl: HNO₃) in a Teflon bomb for 2 h at 120 C°. After

cooling, the samples were filtered and kept in plastic bottles (APHA, 2017). Heavy elements (cadmium, copper, iron, manganese, nickel, lead, and zinc) have been measured using Inductively Coupled Plasma-Emission Spectrometry (ICP-OES). Ammonium (NH₄-N), Nitrate (NO₃-N) and available phosphorus (PO₄-P) was determined spectrophotometric ally using the spectrophotometric as described by APHA, (2017).

Statistical Analysis

Data were analyzed for effects of bentonite and zeolite treatments on stabilize nutrients and heavy metals within earthen pond sediments. Using a one-way MANOVA in SPSS statistics to evaluate the significant differences for each parameter of the nutrient and heavy metal extracted with each treatment during different incubation periods i.e. (3 time periods) 15, 30 and 45 days. Statistical analysis was performed using SPSS 25 statistical program. All significance levels mentioned in the text were $p < 0.05$ (one-way MANOVA in SPSS 25).

RESULTS AND DISCUSSION

Chemical properties of water supply and artificial fish feed

Some chemical properties of water supply for ponds and artificial feeding of grass carp fingerlings were measured as shown in Table (1).

Table 1. Chemical properties of water supply and artificial feeding for grass carp fingerling ponds

Parameters	Water supply (Nile River)	Law 48/1982	Parameters	Artificial fish feed (pellets 25% protein)
pH	8.04	7 - 8.5	-	-
EC dSm ⁻¹	0.42	0.5	-	-
NH ₄ ⁺ mg L ⁻¹	0.07	0.5	-	-
NO ₃ ⁻ mg L ⁻¹	0.17	45	-	-
PO ₄ ³⁻ mg L ⁻¹	0.45	-	-	-
Cd mg L ⁻¹	0.005	0.01	Cd µg/g	2.10
Cu mg L ⁻¹	0.013	1	Cu µg/g	7.62
Fe mg L ⁻¹	0.165	1	Fe µg/g	175.5
Mn mg L ⁻¹	0.115	0.5	Mn µg/g	42.33
Ni mg L ⁻¹	0.008	-	Ni µg/g	6.21
Pb mg L ⁻¹	0.015	0.05	Pb µg/g	3.95
Zn mg L ⁻¹	0.035	1	Zn µg/g	11.23

The results of the water supply in the ponds showed levels lower than the maximum permissible limits of Egyptian Law No. 48/1982. The mean concentrations of heavy elements in the water tended to be in the order of Fe > Mn > Zn > Pb > Cu > Ni > Cd. It is clear that the ability of surface sediment in the earthen ponds to accumulate heavy elements from the water is variable. Concerning the artificial

diet, it had almost the same order except for Pb (Fe > Mn > Zn > Cu > Ni > Pb > Cd), this is in agreement with (Das, *et al.*, 2017) they reported that the metal concentrations in fish feed followed the sequence Cr>Cu>Ni>Pb>Cd.

Chemical properties of the pond sediments after the end of the fingerlings rearing

The results of the sediments showed in the five earthen ponds after the end of the fingerlings rearing as shown in Table (2) showed that the values of pH ranged between 7.48 – 7.73. The electrical conductivity (EC) ranged between 0.628 – 0.652 dSm⁻¹, the EC is an indicator of the content of dissolved inorganic salts in the surface sediment. The concentrations of nutrients ranged between 10.90 – 12.32, 14.50 – 17.25 and 9.80 – 11.38 µg/ g for NH₄⁺, NO₃⁻ and P, respectively. The concentration of heavy elements ranged between 0.059 – 0.082, 0.299 – 0.365, 10.56 – 85.32, 1.98 – 2.35, 0.66 – 0.78 and 7.99 – 9.25 µg/ g for Cd, Cu, Mn, Ni, Pb, and Zn, respectively, except for Fe ranged between 12.35 and 69.81 mg/g. The results of the heavy elements and nutrients in the ponds can be mainly attributed to the feeding process (uneaten, decaying food and faecal wastes from fishes due to food consumption). This interpretation may reinforce that the order of abundance of the heavy elements followed the same trend like the artificial diet. As the feeding waste settle to the bottom and the sediments absorbed these heavy elements and nutrients, the sediments in this case act as a major reservoir for these elements in the aquatic environment. This interpretation of the results is agree with (Ali *et al.*, 2006; Xinhua, 2012; El-Kholy, 2013 and Mansour *et al.*, 2019) reported that heavy elements, nutrients and organic residues usually result from feeding and tend to accumulate at the bottom of the pond, the excessive accumulation in the sediment carrying may result in the deterioration of the pond environment by their releasing into the water column when weakly bound within sediments, with the lower pH of the sediment, facilitating their chemically or biologically mediated release into waters (Kabata-Pendias and Pendias 1992; Blasco, *et al.*, 2000; Osman and Kloas 2010; Yin *et al.*, 2016).

Table 2. Chemical properties (± SD) in the five earthen pond sediments at the end of the stocked period of fingerlings grass carp in DBRS

Parameters	Pond 1 (P1)	Pond 2 (P2)	Pond 3 (P3)	Pond 4 (P4)	Pond 5 (P5)
pH	7.48±0.10	7.58±0.12	7.68±0.08	7.66±0.11	7.73±0.09
EC dS m ⁻¹	0.652±0.009	0.632±0.010	0.621±0.012	0.637±0.006	0.628±0.008
NH ₄ -N µg/ g	12.32±0.29	11.80±0.23	11.60±0.33	12.13±0.19	10.90±0.35
NO ₃ -N µg/ g	17.25±0.28	16.40±0.18	15.50±0.26	14.50±0.22	16.90±0.33
PO ₄ -P µg/ g	11.38±0.15	9.80±0.19	11.20±0.13	10.80±0.21	11.10±0.18
Cd µg/ g	0.082±0.006	0.067±0.003	0.081±0.007	0.059±0.003	0.075±0.007
Cu µg/ g	0.365±0.012	0.321±0.013	0.299±0.009	0.311±0.011	0.345±0.014
Fe mg/ g	12.35±0.25	11.55±0.32	10.56±0.19	11.32±0.24	12.05±0.21
Mn µg/ g	85.32±1.04	77.25±0.89	81.4±1.14	69.81±1.15	78.43±1.19
Ni µg/ g	2.35±0.08	2.11±0.07	1.98±0.10	2.08±0.06	2.22±0.11
Pb µg/ g	0.781±0.035	0.714±0.019	0.689±0.016	0.722±0.028	0.668±0.031
Zn µg/ g	9.25±0.24	8.88±0.27	8.65±0.32	7.99±0.29	8.32±0.34

Through the results presented in Table (2), the sediments of Pond 1 (P1) with the highest values of chemical properties among the five ponds were selected to apply the bentonite and zeolite treatments.

The effectiveness of mixing or capping of bentonite and zeolite for sediment to stabilize nutrients and heavy metals

The effectiveness uses of bentonite and zeolite under two different amendment methods, i.e., mixing and capping,

for stabilize of nutrients and heavy metals in sediment was assessed.

Effect of capping and mixing on pH and EC

The mean pH values of sediments for bentonite and zeolite treatments (BM 4%, BM 6%, BC 4%, BC 6%, ZM 4%, ZM 6%, ZC 4% and ZC 6%) was observed a slight high (from 7.72 to 8.32) compared to control (from 7.65 to 7.76) as shown in Table (3). There were significant differences in pH for all treatments ($p < 0.05$), but not significant for

control. This increase in the environmental pH could promote bentonite and zeolite surface adsorption of metal ions, for bentonite, it can be due to the hydrolysis of montmorillonite (the exchangeable cation of Na replaced with H⁺ from water), while for zeolite, it can be due to the ion exchange between Na⁺ and protons, this agrees with (Usman *et al.*, 2004; Stephan, *et al.*, 2008; Li *et al.*, 2009; Sen *et al.*, 2011; Ali, 2014 and Wen *et al.*, 2016). The results in Table (3) show that slight high in the mean EC concentration in sediments for bentonite and zeolite treatments was observed. This slight high in EC may be due

to the silicates melt emitted by bentonite and zeolite, but the EC concentration during incubation periods in bentonite treatments is lower than in zeolite treatments because the solubility of bentonite silicate is lower than of zeolite silicate. The mean EC concentration in sediments for control was higher than all treatments, this result agrees with Hassan and Mahmoud (2013) who reported that the treated soil by the combined zeolite and bentonite were less saline than the untreated ones. There were significant differences in EC for all treatments ($p < 0.05$), but not significant for control.

Table 3. Effects of capping and mixing of bentonite and zeolite on pH and EC in sediment and the significant variation among them during 45 days of incubation period

Treatments	Incubation period (days)			Sig. (P ≤ 0.05)		
	15	30	45	(15-30)	(15-45)	(30-45)
	PH					
Ctrl.	7.65±0.10	7.72±0.02	7.76±0.04	0.084 ^{NS}	0.069 ^{NS}	0.431 ^{NS}
BM 4%	7.72±0.04	8.08±0.04	8.19±0.04	0.017*	0.008*	0.050*
BM 6%	7.75±0.03	8.10±0.05	8.25±0.02	0.018*	0.001*	0.039*
BC 4%	7.81±0.03	8.13±0.02	8.28±0.02	0.022*	0.009*	0.035*
BC 6%	7.81±0.02	8.15±0.02	8.32±0.02	0.001*	0.018*	0.027*
ZM 4%	7.76±0.03	8.04±0.03	8.19±0.03	0.015*	0.003*	0.048*
ZM 6%	7.80±0.03	8.12±0.03	8.25±0.02	0.003*	0.007*	0.033*
ZC 4%	7.85±0.03	8.12±0.02	8.26±0.02	0.001*	0.001*	0.023*
ZC 6%	7.88±0.03	8.15±0.02	8.28±0.02	0.001*	0.008*	0.001*
	EC (dS m ⁻¹)					
Ctrl.	0.691±0.006	0.695±0.003	0.701±0.004	0.765 ^{NS}	0.968 ^{NS}	0.779 ^{NS}
BM 4%	0.663±0.005	0.666±0.003	0.673±0.004	0.001*	0.023*	0.045*
BM 6%	0.665±0.002	0.672±0.002	0.677±0.003	0.046*	0.001*	0.034*
BC 4%	0.668±0.002	0.669±0.002	0.672±0.005	0.001*	0.012*	0.024*
BC 6%	0.669±0.006	0.671±0.002	0.678±0.004	0.011*	0.036*	0.003*
ZM 4%	0.667±0.002	0.678±0.002	0.687±0.002	0.008*	0.006*	0.045*
ZM 6%	0.670±0.003	0.683±0.002	0.691±0.004	0.001*	0.016*	0.031*
ZC 4%	0.669±0.001	0.681±0.002	0.686±0.002	0.002*	0.001*	0.015*
ZC 6%	0.667±0.001	0.685±0.002	0.691±0.003	0.001*	0.010*	0.008*

NS: Not significant, *P<0.05.

Effect of capping and mixing for bentonite and zeolite on nutrients stabilize in sediments

The results in Table (4) indicate that the use of bentonite and zeolite as capping and mixing with sediments lead to a decrease in the extraction ratio of NH₄-N and NO₃-N from sediments compared to control, and they had a tendency to settle inside the sediments in different percentages. There were significant differences in NH₄-N concentrations for all treatments ($p < 0.05$), but not significant for control. Also, for NO₃-N there are significant differences for all treatments, but not significant for control, BM 4% and BM 6% between (30-45). Generally, the capping or mixing of zeolite with sediments had significantly for interrupting the release of NH₄-N and NO₃-N from sediments compared to the capping or mixing of bentonite (P<0.05). Also, capping by zeolite was found to be more effective than mixing. The decrease in extractability of NH₄-N from sediments owing to bentonite and zeolite applications seemed to be in the following descending order: ZC 6% > ZC 4% > ZM 6% > ZM 4% > BC 6% > BC 4% > BM 6% > BM 4%. Also in the same direction, the decrease in extractability of NO₃-N from sediments owing to bentonite and zeolite applications seemed to be in the following descending order: ZC 6% > ZC 4% > ZM 6% > ZM 4% > BC 6% > BC 4% > BM 6% > BM 4%.

The results shown in Table (4) indicate that the use of bentonite and zeolite as capping and mixing with sediments lead to a decrease in the extraction ratio of PO₄-P

from sediments compared to control, and they had a tendency to settle inside the sediments in different percentages. There were significant differences in PO₄-P concentrations for all treatments ($p < 0.05$), but not significant for control, ZM 4% and ZM 6% between (30-45). Generally, the capping or mixing of bentonite with sediments had significantly higher for interrupting the release of PO₄-P from sediments compared to the capping or mixing of zeolite (P<0.05). Also, capping by bentonite was found to be more effective than mixing. The decrease in extractability of PO₄-P from sediments owing to bentonite and zeolite applications seemed to be in the following descending order: BC 6% > BC 4% > BM 6% > BM 4% > ZC 6% > ZC 4% > ZM 6% > ZM 4%.

The efficiency of the bentonite and zeolite treatments for stabilizing NH₄-N, NO₃-N and PO₄-P was calculated compared to the non-treatment as shown in Table (4) and Figure (2).The zeolite treatments were more efficient for stabilize of NH₄-N and NO₃-N in sediments than the bentonite treatments. While, bentonite treatments were more efficient for stabilize PO₄-P in sediments than the zeolite treatments. Also, the capping of materials by bentonite and zeolite on the sediment surface for the NH₄-N, NO₃-N and PO₄-P stabilizing was more efficient than mixing the materials with the sediment for the same treatment. ZC 6% have the highest decrease in extractability ratio for NH₄-N (65.4%) and NO₃-N (41.8%), while, BM

4% have the lowest decrease in extractability ratio for NH₄-N (27.3 %) and NO₃-N (10.7 %). Also, BC 6% have the highest decrease in extractability ratio for PO₄-P (61.9 %),

while, BM 4% have the lowest decrease in extractability ratio for PO₄-P (7.3 %).

Table 4. Effects of capping and mixing of bentonite and zeolite on extractability of NH₄-N, NO₃-N and PO₄-P from sediment and their decrease percentages and the significant variation among them during 45 days of incubation period

Treatments	Incubation period (days)			% of decrease			Sig. (P ≤ 0.05)		
	15	30	45	15	30	45	(15-30)	(15-45)	(30-45)
NH ₄ -N (µg/g)									
Ctrl.	12.09±0.17	12.12±0.23	12.36±0.19	-	-	-	0.792 ^{NS}	0.055 ^{NS}	0.089 ^{NS}
BM 4%	11.29±0.17	9.51±0.21	8.99±0.21	6.6	21.5	27.3	0.006*	0.018*	0.031*
BM 6%	10.77±0.20	9.28±0.13	8.56±0.16	10.9	23.5	30.7	0.009*	0.003*	0.041*
BC 4%	10.18±0.14	8.76±0.13	8.18±0.12	15.7	27.7	33.8	0.013*	0.008*	0.027*
BC 6%	9.81±0.22	8.20±0.22	7.41±0.19	18.9	32.4	40.0	0.002*	0.007*	0.036*
ZM 4%	10.13±0.17	6.93±0.25	5.98±0.15	16.2	42.8	51.6	0.009*	0.014*	0.008*
ZM 6%	9.56±0.22	6.29±0.26	5.62±0.19	20.9	48.2	54.5	0.024*	0.005*	0.017*
ZC 4%	9.01±0.13	6.03±0.13	4.98±0.20	25.5	50.3	59.7	0.001*	0.003*	0.021*
ZC 6%	8.73±0.14	5.78±0.21	4.27±0.22	27.8	52.3	65.4	0.0010*	0.017*	0.004*
NO ₃ -N (µg/g)									
Ctrl.	7.10±0.23	7.13±0.07	6.72±0.13	-	-	-	0.752 ^{NS}	0.122 ^{NS}	0.091 ^{NS}
BM 4%	6.43±0.18	6.37±0.23	6.01±0.10	9.4	10.7	10.7	0.007*	0.003*	0.604 ^{NS}
BM 6%	5.98±0.16	6.32±0.23	5.84±0.12	15.8	11.4	13.2	0.008*	0.001*	0.222 ^{NS}
BC 4%	5.83±0.12	6.31±0.14	5.49±0.11	18.0	11.5	18.4	0.017*	0.001*	0.038*
BC 6%	5.69±0.16	6.28±0.37	5.02±0.22	19.8	12.0	25.3	0.005*	0.005*	0.003*
ZM 4%	6.47±0.27	5.95±0.45	4.58±0.19	23.0	16.7	31.9	0.036*	0.028*	0.001*
ZM 6%	5.13±0.15	5.66±0.11	4.50±0.19	27.8	20.7	33.1	0.000*	0.009*	0.019*
ZC 4%	4.76±0.32	5.64±0.13	4.24±0.14	30.0	21.0	37.0	0.006*	0.003*	0.031*
ZC 6%	4.32±0.19	5.20±0.29	3.91±0.10	39.1	27.0	41.8	0.007*	0.009*	0.027*
PO ₄ -P (µg/g)									
Ctrl.	10.97±0.15	10.83±0.19	10.74±0.22	-	-	-	0.274 ^{NS}	0.070 ^{NS}	0.148 ^{NS}
BM 4%	9.55±0.38	7.68±0.21	6.79±0.37	12.9	29.0	36.2	0.005*	0.003*	0.049*
BM 6%	8.41±0.25	6.96±0.21	6.15±0.18	23.3	35.8	42.2	0.009*	0.001*	0.035*
BC 4%	8.22±0.11	6.21±0.16	5.14±0.27	25	42.7	51.7	0.030*	0.008*	0.009*
BC 6%	8.13±0.12	5.65±0.17	4.06±0.12	25.9	47.8	61.9	0.006*	0.003*	0.007*
ZM 4%	10.75±0.15	10.16±0.25	9.87±0.14	2.0	6.2	7.3	0.010*	0.029*	0.132 ^{NS}
ZM 6%	10.59±0.18	9.75±0.17	9.12±0.38	3.4	9.9	14.3	0.008*	0.002*	0.068 ^{NS}
ZC 4%	9.91±0.27	9.23±0.16	8.79±0.25	9.6	14.8	17.4	0.041*	0.013*	0.011*
ZC 6%	9.60±0.26	8.77±0.17	8.27±0.25	12.4	19.0	22.3	0.007*	0.008*	0.005*

% of decrease: Calculated by comparing each treatment to Ctrl.
NS: Not significant, *P< 0.05.

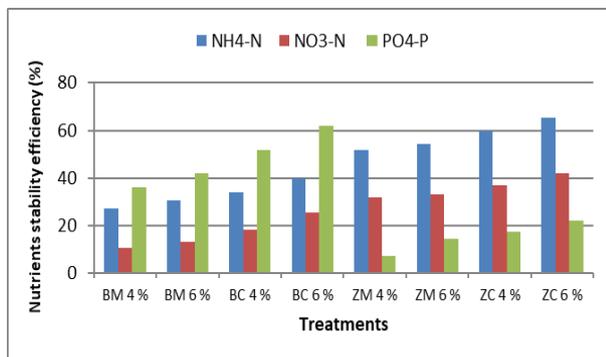


Fig. 2. Comparison of treatments efficiency for capping and mixing of bentonite and zeolite for stabilization of NH₄-N, NO₃-N and PO₄-P in sediment

Similar results were found in previous studies for effect of capping and mixing using bentonite and zeolite on stabilize of NH₄-N, NO₃-N and PO₄-P in sediments. Miltiadis *et. al.*, (2015); Gu, *et. al.*, (2019) stated that bentonite and zeolite as capping materials through their high adsorption capacity prevent the flux phosphorus and ammonium from eutrophic lake sediments and interrupt their release into water. Chunhui *et. al.*, (2018) demonstrated that the efficiency of sediment capping using active thin-layer capping with natural zeolite with 2 cm thickness, resulted in the inhibition of NH₄⁺ and PO₄³⁻. Zeolite was proven to be effective for interrupting the release of nitrogen (NH₄-N, NO₃-N) from contaminated

lake sediment to the water (Alvarado *et. al.*, 2020). Ali, (2014) obtained that the use of bentonite at 4% (w/w) as mixing materials with earthen pond sediments used in raising grass carp breeders was reduced NH₄-N, NO₃-N and P extraction.

Effect of capping and mixing for bentonite and zeolite on stabilize of heavy metals in sediments

The presented results in Table (5) indicate that the use of bentonite and zeolite as capping and mixing with sediments lead to a decrease in the extraction ratio of Cd, Cu, Pb and Zn from sediments compared to control, and they had a tendency to settle inside the sediments in different percentages. There were significant differences in Cd, Cu, Pb and Zn concentrations for all treatments (p < 0.05), but not significant both for control, and BM 4%, BM 6% and Bc 4% treatment between (30-45) for Cd and Pb. Generally, the capping or mixing of zeolite with sediments for interrupting the release of Cd, Cu, Pb and Zn from sediments were significantly (P<0.05) higher than capping or mixing of bentonite with sediments. Also, capping by zeolite was found to be more effective than mixing. The decrease in extractability of Cd, Cu, Pb and Zn from sediments owing to bentonite and zeolite applications seemed to be in the following descending order: ZC 6% > ZC 4% > ZM 6% > ZM 4% > BC 6% > BC 4% > BM 6% > BM 4%. Heavy metals uptake could mainly be attributed to the adsorption on the surface of the zeolite as a result the ion-exchange reactions in the micro porous minerals of the zeolite (such,

mainly sodium, calcium and smaller potassium), as well as the increase in pH. This interpretation agrees with (Curkovic *et al.*, 1997). Also, these results agree with (Castaldi *et al.*, 2005; Mahabadi *et al.*, 2007; Humidpour *et al.*, 2010; Jia *et al.*, 2016; Chunhui *et al.*, 2018) showed that adding zeolite to soil contaminated with Cd, Cu, Pb and Zn compounds led to a stronger immobilizing of these minerals and reduced their concentrations in the soil, it was the most suitable for Cd stabilization.

The presented results in Table (6) indicate that the use of bentonite and zeolite as capping and mixing with sediments lead to a decrease in the extraction ratio of Fe, Mn and Ni from sediments compared to control, and they had a tendency to settle inside the sediments in different percentages. There were significant differences in Fe, Mn and Ni concentrations for all treatments ($p < 0.05$), but not significant both for control, and ZM 4% treatment between (30-45) for Fe, Mn and Ni. Generally, the capping or mixing

of bentonite with sediments for interrupting the release of Fe, Mn and Ni from sediments were significantly ($P < 0.05$) higher than capping or mixing of zeolite with sediments. Also, capping by bentonite was found to be more effective than mixing. The decrease in extractability of Fe, Mn and Ni from sediments owing to bentonite and zeolite applications seemed to be in the following descending order: BC 6% > BC 4% > BM 6% > BM 4% > ZC 6% > ZC 4% > ZM 6% > ZM 4%. Heavy metals uptake could be attributed to the adsorption on the surface of the bentonite due to the increase in pH and great ability of the bentonite to absorb cations in its micro pores. These results agree with Usman *et al.*, (2004) who found that addition of bentonite decreased the extractability of heavy metals during incubation. Also, agree with Ali, (2014) who obtained that the use of bentonite at 4% (w/w) as mixing materials with earthen pond sediments used in raising grass carp breeders was reduced Cu, Mn, Fe, Ni, Zn, Cd and Pb extraction, respectively.

Table 5. Effects of capping and mixing of bentonite and zeolite on extractability of Cd, Cu, Pb and Zn from sediment and their decrease percentages and the significant variation among them during 45 days of incubation period

Treatments	Incubation period (days)			% of decrease			Sig. ($P \leq 0.05$)		
	15	30	45	15	30	45	(15-30)	(15-45)	(30-45)
Cd ($\mu\text{g/g}$)									
Ctrl.	0.079±0.001	0.076±0.002	0.074±0.002	-	-	-	0.058 ^{NS}	0.163 ^{NS}	0.131 ^{NS}
BM 4%	0.068±0.002	0.063±0.004	0.059±0.001	13.3	16.4	20.1	0.024*	0.041*	0.087 ^{NS}
BM 6%	0.066±0.002	0.058±0.002	0.055±0.002	15.9	23.3	25.0	0.016*	0.009*	0.142 ^{NS}
BC 4%	0.066±0.004	0.056±0.003	0.052±0.002	16.2	25.7	28.8	0.001*	0.010*	0.085 ^{NS}
BC 6%	0.063±0.001	0.054±0.002	0.048±0.004	20.4	28.3	35.3	0.002*	0.028*	0.005*
ZM 4%	0.059±0.004	0.039±0.003	0.032±0.002	25.5	48.1	56.3	0.024*	0.009*	0.032*
ZM 6%	0.056±0.003	0.035±0.003	0.030±0.004	29.0	53.7	59.5	0.006*	0.001*	0.033*
ZC 4%	0.054±0.002	0.033±0.002	0.027±0.003	31.8	55.8	62.8	0.006*	0.021*	0.036*
ZC 6%	0.051±0.002	0.030±0.002	0.025±0.003	35.0	60.3	66.0	0.037*	0.022*	0.006*
Cu ($\mu\text{g/g}$)									
Ctrl.	0.348±0.005	0.344±0.008	0.342±0.008	-	-	-	0.342 ^{NS}	0.176 ^{NS}	0.661 ^{NS}
BM 4%	0.291±0.007	0.247±0.008	0.212±0.008	16.6	28.2	38.1	0.003*	0.017*	0.048*
BM 6%	0.279±0.001	0.211±0.008	0.183±0.006	20.0	38.7	46.6	0.008*	0.014*	0.050*
BC 4%	0.269±0.007	0.197±0.004	0.176±0.010	22.8	42.7	48.5	0.027*	0.005*	0.026*
BC 6%	0.266±0.006	0.185±0.010	0.161±0.004	23.7	46.1	52.9	0.003*	0.006*	0.041*
ZM 4%	0.265±0.008	0.182±0.009	0.159±0.006	23.9	47.2	53.5	0.012*	0.001*	0.031*
ZM 6%	0.264±0.005	0.173±0.007	0.149±0.006	24.2	49.7	56.4	0.008*	0.002*	0.022*
ZC 4%	0.256±0.004	0.156±0.004	0.136±0.009	26.5	54.4	60.4	0.014*	0.001*	0.005*
ZC 6%	0.234±0.004	0.143±0.007	0.123±0.009	32.8	58.4	63.9	0.009*	0.003*	0.012*
Pb ($\mu\text{g/g}$)									
Ctrl.	0.732±0.027	0.722±0.019	0.708±0.024	-	-	-	0.520 ^{NS}	0.138 ^{NS}	0.372 ^{NS}
BM 4%	0.646±0.027	0.560±0.016	0.536±0.015	11.7	22.4	24.3	0.001*	0.005*	0.143 ^{NS}
BM 6%	0.630±0.035	0.520±0.016	0.488±0.026	13.9	27.9	31.1	0.002*	0.016*	0.084 ^{NS}
BC 4%	0.582±0.019	0.490±0.016	0.472±0.031	20.5	32.1	33.3	0.002*	0.025*	0.240 ^{NS}
BC 6%	0.566±0.020	0.482±0.019	0.454±0.015	22.7	33.2	35.9	0.018*	0.007*	0.034*
ZM 4%	0.560±0.022	0.472±0.015	0.424±0.011	23.5	34.6	40.1	0.023*	0.009*	0.021*
ZM 6%	0.544±0.021	0.454±0.021	0.376±0.027	25.7	37.1	46.9	0.023*	0.001*	0.045*
ZC 4%	0.528±0.018	0.430±0.032	0.368±0.019	27.9	40.4	48.0	0.009*	0.032*	0.006*
ZC 6%	0.482±0.019	0.380±0.022	0.324±0.024	34.2	47.4	54.2	0.023*	0.008*	0.002*
Zn ($\mu\text{g/g}$)									
Ctrl.	8.74±0.17	8.38±0.12	8.28±0.12	-	-	-	0.068 ^{NS}	0.125 ^{NS}	0.263 ^{NS}
BM 4%	7.62±0.29	6.59±0.72	5.80±0.27	12.7	21.4	29.9	0.005*	0.003*	0.021*
BM 6%	7.56±0.24	6.08±0.10	5.44±0.13	13.5	27.5	34.3	0.033*	0.014*	0.029*
BC 4%	7.46±0.20	5.75±0.16	5.21±0.16	14.6	31.4	37.1	0.005*	0.013*	0.049*
BC 6%	7.30±0.17	5.54±0.16	4.61±0.18	16.5	33.9	44.3	0.011*	0.008*	0.022*
ZM 4%	7.22±0.10	5.38±0.12	4.52±0.06	17.4	35.7	45.4	0.009*	0.001*	0.038*
ZM 6%	7.10±0.08	5.16±0.16	4.23±0.10	18.7	38.4	48.9	0.005*	0.013*	0.042*
ZC 4%	7.02±0.05	4.87±0.07	3.93±0.07	19.7	41.9	52.5	0.008*	0.003*	0.023*
ZC 6%	6.74±0.19	4.67±0.19	3.54±0.18	22.9	44.3	57.3	0.010*	0.001*	0.001*

% of decrease: Calculated by comparing each treatment to Ctrl.

NS: Not significant, * $P < 0.05$.

Table 6. Effects of capping and mixing of bentonite and zeolite on extractability of Fe, Mn and Ni from sediment and their decrease percentages and the significant variation among them during 45 days of incubation period

Treatments	Incubation period (days)			% of decrease			Sig. (P ≤ 0.05)		
	15	30	45	15	30	45	(15-30)	(15-45)	(30-45)
	Fe (mg/g)								
Ctrl.	12.04±0.07	11.81±0.11	11.69±0.20	-	-	-	0.124 ^{NS}	0.202 ^{NS}	0.372 ^{NS}
BM 4%	10.68±0.25	8.63±0.09	8.04±0.07	11.3	26.9	31.3	0.011*	0.003*	0.046*
BM 6%	10.32±0.15	8.38±0.13	7.54±0.19	14.3	29.1	35.5	0.024*	0.001*	0.008*
BC 4%	10.14±0.18	8.20±0.09	7.40±0.13	15.8	30.6	36.7	0.032*	0.004*	0.048*
BC 6%	9.99±0.23	8.06±0.08	6.71±0.17	17.0	31.8	42.7	0.001*	0.003*	0.039*
ZM 4%	11.14±0.16	9.69±0.18	9.01±0.09	7.5	17.9	23.0	0.041*	0.005*	0.089 ^{NS}
ZM 6%	11.01±0.18	9.43±0.19	8.52±0.21	8.6	20.2	27.2	0.003*	0.001*	0.037*
ZC 4%	10.93±0.17	9.14±0.13	8.33±0.06	9.2	22.6	28.8	0.022*	0.008*	0.031*
ZC 6%	10.69±0.22	8.68±0.09	8.05±0.12	11.2	26.5	31.2	0.009*	0.005*	0.001*
	Mn (µg/g)								
Ctrl.	81.64±0.53	81.43±2.04	79.35±1.37	-	-	-	0.826 ^{NS}	0.129*	0.243*
BM 4%	68.50±0.56	53.47±1.51	47.37±1.34	16.1	34.3	40.3	0.033*	0.001*	0.013*
BM 6%	67.29±0.50	48.78±0.70	42.16±1.47	17.6	40.1	46.9	0.009*	0.002*	0.045*
BC 4%	65.86±0.46	46.79±1.09	38.25±0.46	19.3	42.5	51.8	0.012*	0.008*	0.001*
BC 6%	63.99±1.01	42.54±0.75	34.11±0.87	21.6	47.8	57.0	0.022*	0.001*	0.047*
ZM 4%	73.96±0.84	61.70±0.88	57.18±0.73	9.4	24.2	27.9	0.008*	0.011*	0.079 ^{NS}
ZM 6%	72.65±0.47	61.25±0.39	54.29±0.85	11.0	24.8	31.6	0.002*	0.000*	0.042*
ZC 4%	71.39±0.86	58.51±0.40	52.53±0.52	12.6	28.2	33.8	0.005*	0.007*	0.050*
ZC 6%	70.33±0.71	55.46±0.36	49.23±1.20	13.9	31.9	37.9	0.013*	0.001*	0.029*
	Ni (µg/g)								
Ctrl.	2.22±0.02	2.20±0.02	2.18±0.03	-	-	-	0.309 ^{NS}	0.066 ^{NS}	0.110 ^{NS}
BM 4%	1.91±0.09	1.45±0.05	1.28±0.05	13.8	34.3	41.1	0.041*	0.016*	0.002*
BM 6%	1.81±0.08	1.34±0.14	1.18±0.07	18.5	39.2	45.8	0.013*	0.008*	0.028*
BC 4%	1.79±0.06	1.31±0.07	1.11±0.07	19.5	40.4	49.0	0.002*	0.010*	0.001*
BC 6%	1.71±0.08	1.29±0.11	1.03±0.06	23.2	41.3	52.6	0.014*	0.003*	0.033*
ZM 4%	2.07±0.05	1.73±0.09	1.59±0.08	6.8	21.3	27.1	0.026*	0.005*	0.782 ^{NS}
ZM 6%	2.03±0.04	1.69±0.07	1.51±0.08	8.4	23.4	30.8	0.006*	0.001*	0.021*
ZC 4%	1.93±0.06	1.47±0.10	1.44±0.19	13.2	33.4	31.8	0.001*	0.026*	0.042*
ZC 6%	1.91±0.08	1.48±0.10	1.39±0.08	14.1	33.0	36.3	0.005*	0.038*	0.045*

% of decrease: Calculated by comparing each treatment to Ctrl.
 NS: Not significant, *P<0.05.

The efficiency of the bentonite and zeolite treatments for stabilizing heavy metals was calculated compared to the non-treatment as shown in Tables (5, 6) and Figure (3).

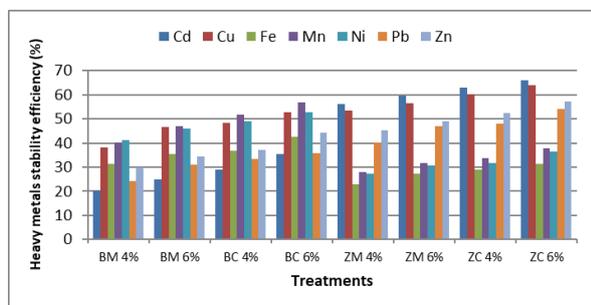


Fig. 3. Comparison of treatments efficiency for capping and mixing of bentonite and zeolite for stabilization of heavy metals in sediment

The zeolite treatments were more efficient for stabilize of Cd, Cu, Pb and Zn in sediments than the bentonite treatments. While, bentonite treatments were more efficient for stabilize Fe, Mn and Ni in sediments than the zeolite treatments. The capping of bentonite or zeolite on the surface of the sediment was more efficient for interrupting the release of heavy metals than the same treatment mixing the bentonite or zeolite with the sediment. ZC 6% have the highest decrease in extractability ratio for Cd (66.0 %), Cu (63.9%), Pb (54.2 %) and Zn (57.3 %), while, BM 4% have the lowest decrease in extractability ratio for Cd (20.1 %), Cu (38.1 %), Pb (24.3 %) and Zn (29.9 %). BC 6% have the highest decrease in extractability ratio for Fe (42.7 %), Mn (57.0 %) and Ni (52.6 %), while, ZM 4% have the lowest decrease in extractability ratio for

Fe (23.0 %), Mn (27.9 %) and Ni (27.1 %). The explanation for the low extractability can be attributed to the increased pH of the sediments, and zeolite and bentonite have high CEC that exchange of heavy metals can occur in the structure of zeolite and bentonite. The stabilization efficiency of Cu, Ni, Zn, Cd and Pb by bentonite is largely consistent with the results bottomed by Karapinar and Donat, 2009; Bertagnolli *et al.*, 2011; Neto *et al.*, 2012; Hannachi *et al.*, 2013; Ali, 2014 and Oupa and Kapil, 2018. While, the stabilization efficiency of Cd, Pb, Cu, Mn and Zn by zeolite is consistent with the results of Castaldi, *et al.*, 2005; Mahabadi, *et al.*, 2007; Li, *et al.*, 2009; Humidpour, *et al.*, 2010; Jia, *et al.*, 2016; Wen, *et al.*, 2016 and Chunhui, *et al.*, 2018.

In general, effect of capping and mixing for bentonite and zeolite on nutrients stabilize in sediments during incubation periods, an increase in the absorbed amount of nutrients and heavy metals was observed with increasing incubation period (contact time) for both bentonite and zeolite 4%, 6% whether covering or mixing as shown in Tables (4, 5 and 6).

CONCLUSION AND RECOMMENDATION

In this research, the efficiency of uses of bentonite and zeolite in different percentages was evaluated under two different amendment methods (capping and mixing) to stabilize NH₄-N, NO₃-N, PO₄-P and heavy metal in sediments for interrupt their release into water. In general, an increase in the absorbed amount of nutrients and heavy metals was observed with an increase in the percentage of bentonite and zeolite added, contact time, and pH. The stabilization of nutrients and heavy metals within earthen pond sediments is attributed to the high cation exchange

capacity (CEC) of bentonite and zeolite, which causes the precipitation of these minerals within the bentonite or zeolite, and prevents their release from the sediments to the water. A higher slight pH and EC was observed in sediments that capped or mixed with bentonite and zeolite. The highest stabilization efficiency of $\text{NH}_4\text{-N}$, $\text{NO}_3\text{-N}$, Cd, Cu, Pb and Zn in the sediments was recorded with ZC 6% by 65.4%, 41.8%, 66.0 %, 63.9 %, 54.2 % and 57.3 %, respectively, while, the highest stabilization efficiency of $\text{PO}_4\text{-P}$, Fe, Mn and Ni was with BC 6% by 61.9%, 42.7 %, 57.0 % and 52.6 %, respectively. Capping treatments was better than mixing for the treatments of bentonite and zeolite in order to stabilize of $\text{NH}_4\text{-N}$, $\text{NO}_3\text{-N}$, $\text{PO}_4\text{-P}$ and heavy metal in sediments. Therefore, it is recommended to use bentonite or zeolite as capping material for sediment at a rate of 6% at the bottom of the pond can be effective for the control of nutrients and heavy metals when they are increased in earthen pond sediments for interrupt their release into water. Thus, this amendment can be improve water quality and reduce the rate of its change, which contributes to providing good environmental condition for fish in order to obtain a good result from the fish stocking in the earthen pond.

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استخدام البنتونيت والزيوليت لتثبيت المغذيات والمعادن الثقيلة داخل رسوبيات الأحواض الترابية ياسر محمود علي

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إن ترك رسوبيات الأحواض الترابية لعدة سنوات دون تطهير أو معالجة يؤدي إلى وجود تركيزات عالية من مركبات النيتروجين والفوسفات والمعادن الثقيلة، زيادة ملوثات الرسوبيات تجعلها تنطلق في الماء. لذلك، هدف هذا البحث إلى تقييم فعالية تغطية أو خلط البنتونيت والزيوليت بنسب مختلفة لتثبيت المغذيات والمعادن الثقيلة في رسوبيات الأحواض الترابية لعرقلة إطلاقها في الماء. تم قياس الأس الهيدروجيني (pH) والتوصيل الكهربائي (EC) والأمونيوم (NH₄-N) والنترات (NO₃-N) والفوسفات (PO₄-P) والمعادن الثقيلة (الكاديوم Cd، النحاس Cu، الحديد Fe، المنجنيز Mn، النيكل Ni، الرصاص Pb والزنك Zn) في الرسوبيات بتجارب الحضانة المعملية لمدة ٤٥ يوماً. أظهرت النتائج أن أعلى كفاءة تثبيت للـ NO₃-N، NH₄-N، Cu، Cd، Pb و Zn في الرسوبيات سُجلت مع تغطية الزيوليت للرسوبيات بنسبة ٦ % (ZC 6%) حيث بلغت ٦٥،٤ %، ٤١،٨ %، ٦٦،٠ %، ٦٣،٩ %، ٥٤،٢ % و ٥٧،٣ % على التوالي، بينما أعلى كفاءة تثبيت للـ PO₄-P، Fe، Mn و Ni سُجلت مع تغطية البنتونيت للرسوبيات بنسبة ٦ % (BC 6%) حيث بلغت ٦١،٩ %، ٤٢،٧ %، ٥٧،٠ % و ٥٢،٦ % على التوالي. لذلك، يوصى باستخدام البنتونيت أو الزيوليت كمادة تغطية للرسوبيات بمعدل ٦ % في قاع الأحواض يمكن أن يكون فعالاً للتحكم في المغذيات والمعادن الثقيلة عند زيادتها في رسوبيات الأحواض الترابية لعرقلة إطلاقها في الماء.