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In-situ detoxification of soil cadmium- Analysing the potential of LDH composite as an adsorbent

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Abstract. One of the key environmental issues is heavy metal contamination whereby cadmium (Cd) is one of the toxic and long-lasting pollutants of industrial effluents and farm soil. Cadmium was selected to investigate on because it is very mobile, bio accumulative and has a very serious effect on human health and the ecosystem even at low levels. In the present research the possibility of Layered Double Hydroxide (LDH) and LDH/Biochar (LDH/BC) composite derived from rice husk biochar was used as cadmium immobilization agent. The findings showed that cadmium concentration reduced by 16.67% with LDH and 25.8% with LDH/BC treatment, indicating improved immobilization by the composite material. Quantitative microbial analysis after treatment showed higher beneficial bacteria growth over the pathogenic ones. This work identifies LDH-based materials to be a promising and green adsorbent to be used in the removal of heavy metals and provide understanding on the use of the same in the purification of the soil in a sustainable manner.

1. Introduction

Heavy metals are metallic elements with high atomic weights and density that can be toxic even at low concentrations such as Lead (Pb), Cadmium (Cd), Arsenic (As), Nickel (Ni). These metals have a usual density above 5 g/cm^3, which can enter the environment through both natural and anthropogenic processes. Among these Cadmium (Cd) is a very toxic heavy metal which is easily accumulated by promotional industrial effluents, agricultural inputs and waste disposition into soils and it is deadly to the environment and human health [1,2,3]. Because of its perseverance and mobility in earth-crops, efficient methods of reducing the movement and exposure of cadmium is needed. Conventional removal techniques[1] like chemical precipitation and ion exchange methods can be costly and can generate secondary pollutants. Therefore, In-situ fixation by means of soil modification has become a viable and affordable substitute to traditional remediation strategies, since it causes less movement of metal, and does it without altering the framework of the soil.

Layered double hydroxides (LDHs), often described as hydrotalcite-like anionic clays with the general formula $M^{2+}_{1-x}M^{3+}_x(OH)_2.A^{n-}_{x/n}.zH_2O$, where M^{2+} is a metal (II) cation, M^{3+} is metal (III) cation and A^{n-} is an anion [4]. These are crystal structures with high surface areas, interlayer anion exchange and changeable metal composition, and the high amount of -OH groups on the surface of the LDH promotes its binding affinity, thus these structures make a good adsorbent for metal

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ions [5]. However, LDHs may agglomerate quickly during the reaction process, which reduces the surface areas and consequently lowers their adsorption capacity for heavy metals reducing their effectiveness. [6]. This can be tackled by the use of LDH along with Biochar, which is a porous carbon rich material produced by biomass pyrolysis and it has been widely applied in soil remediation due to its adsorbent properties. The combined advantages of biochar and LDH together which may show synergistic effects [7]. Biochar reduces the agglomeration effect of the LDH and improves separation while the LDH contributes to targeted ion-exchange and precipitation mechanisms.

2. Materials and Methods

2.1. Collection of soil sample

Soil samples were collected from agricultural land from the university campus at a depth of approximately 2 inches below the surface after removing visible debris and plant residues. Stones, visible debris and plant remains were cleared manually. The collected soil was homogenized and distributed into four experimental groups, with 4 kg of soil allocated per pot, and each treatment prepared in triplicate.

2.2. Preparation of LDH and LDH/Biochar

Co-precipitation-based method was used in the synthesis of layered double hydroxide (LDH) and LDH/ biochar composites. First of all, rice husks were thoroughly soaked in water, dried in the oven and turned into biochar by controlled pyrolysis at low oxygen and then cooled, ground slightly, and stored. LDH was synthesized from the right precursor salts of magnesium and alkali was used and dissolved in distilled water to prepare a homogeneous precursor, which was then alkalized precisely to cause formation of LDH crystals, it was then isolated. LDH/ biochar composites was prepared using the rice husk biochar with the LDH precursor solution and co-precipitation was induced under controlled alkaline conditions whereby LDH layers were able to nucleate and settle on the biochar surface. The resultant composite was dried, cleaned, and strained to get LDH/ biochar material to be used.

2.3. Preparation of Soil Samples

The collected soil samples were divided into four different groups:

Table 1. Experimental Soil Treatments and Addendum Conditions

Soil Treatment	Description
Control Soil	Soil without cadmium contamination and without any amendment, used as the baseline reference.
Cadmium-Contaminated Soil	4kg of Soil contaminated with cadmium salt at a concentration of 12 µg/kg and aged under open conditions for 7 days.
Cd + LDH Treated Soil	4kg of Cadmium-contaminated soil treated with 2% LDH and aged for 21 days under open conditions
Cd + LDH/Biochar Treated Soil	4kg of Cadmium-contaminated soil treated with 2% LDH/biochar composite and aged for 21 days under open conditions.

The cadmium level (12 mg/kg) chosen was to reflect a moderate level of contamination in soil which is the same as the level of polluted agricultural soils and is the typical level used in laboratory-level remediation studies [8]. The amendment dosage of 2% (w/w) LDH and LDH/Biochar was selected by considering the past research where effective immobilization of heavy metals was observed within the range of 1-5 per cent amendment of soil physicochemical characteristics without causing a radical change on soil physicochemical characteristics.[9]

2.4. Analysis of Soil Samples on Chemical Parameters

The detailed physicochemical assessment of all the soil samples namely uncontaminated control soil, cadmium-contaminated soil, and the treated soils which were amended with LDH and LDH/ biochar composites was done. The determination of total organic carbon was done in accordance with the standard of the determination of soil organic matter as stipulated in the IS 2720: Part 22: 1972 (Reaffirmed 2020). Total nitrogen content was determined using the Kjeldahl method, which involves acid digestion, distillation, and subsequent titration. The cadmium level of each sample was identified by the U.S. EPA Method 3050B, which is an acid-digestion procedure that is used in sediments, sludges, and soils to ensure that the heavy-metal residues were accurately quantified. All the sample groups, namely the control, cadmium-contaminated, LDH-treated, and LDH/biochar-treated soils, had their assessments conducted uniformly in order to determine the effect of cadmium contamination, and to compare the level of remediation by the two adsorbent treatments.

2.5. Analysis of Soil Samples on Microbial Parameters

Microbial analysis of all the soil samples were carried out using culture-based techniques under the same incubation conditions. Serial dilution and plate count techniques were used to determine the number of colonies in Colony Forming Unit (CFU). Standard biochemical tests and Gram staining were further used to identify the respective colonies. The microbes were classified based on the reported ecological roles off identified genera.

2.6. Statistical Analysis

All experiments were conducted in triplicate (n=3). The data is expressed as Mean ± Standard Deviation. Statistical significance was determined using a two-sample t-test assuming unequal variances in Microsoft Excel, with a significance level set at $p<0.05$, $p<0.01$, $p<0.001$.

3. Results

3.1. Soil pH

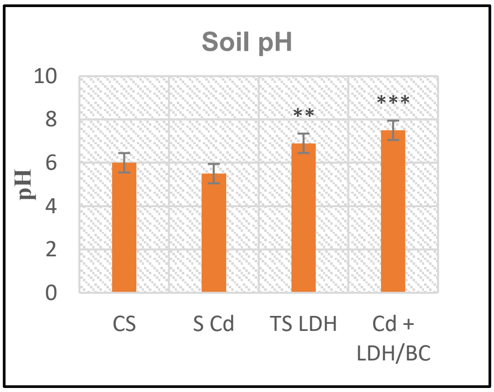

Fig. 1. Soil pH of control and treated Soils. (Data are presented as Mean ± SD (n=3). Asterisks indicate statistical significance compared to the cadmium-contaminated control soil using a two-sample t-test (** $p < 0.01$; *** $p < 0.001$; ns = non-significant)

The control soil with a pH of 6.2 which was slightly acidic. LDH amendment increased the soil pH to 6.9, while LDH/Biochar treatment further elevated it to 7.5, indicating a shift toward near-neutral to mildly alkaline conditions within an acceptable agronomic range.

3.2. Soil Organic Carbon

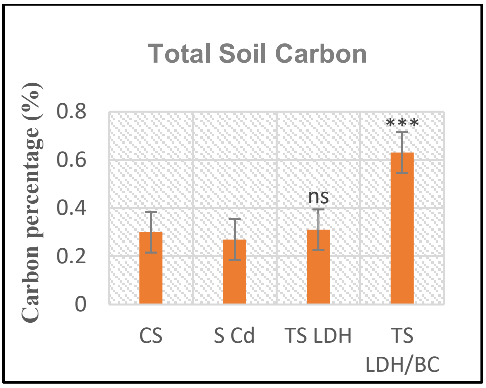

Fig. 2. TOC (%) after LDH and LDH/BC Treatment. (Data are presented as Mean ± SD (n=3). Asterisks indicate statistical significance compared to the cadmium-contaminated control soil using a two-sample t-test (** $p < 0.01$; *** $p < 0.001$; ns = non-significant)

The Control soil had 0.30% Soil Organic Carbon and there was no significant increase in soil organic carbon in the soil with LDH treatment which was 0.31%. In LDH/Biochar-treated soil, the carbon levels increased to 0.63% which was a huge jump in the organic Carbon Concentration levels.

3.3. Soil Nitrogen.

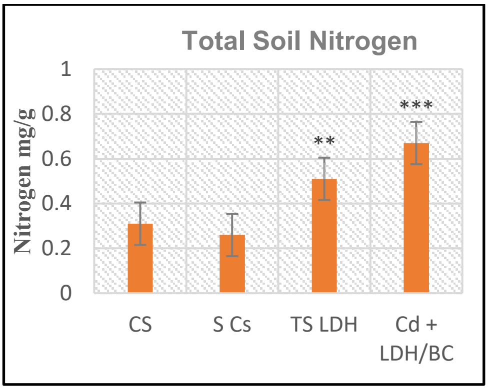

Fig. 3. Total Soil Nitrogen in Control and treated sample. (Data are presented as Mean ± SD (n=3). Asterisks indicate statistical significance compared to the cadmium-contaminated control soil using a two-sample t-test (** $p < 0.01$; *** $p < 0.001$; ns = non-significant)

The level of nitrogen rose in the LDH treated soil from 0.31mg/g to 0.51mg/g. The maximum concentration of nitrogen was 0.67 mg/g which was observed in LDH/Biochar treated soil.

3.4. Soil Cadmium Concentration.

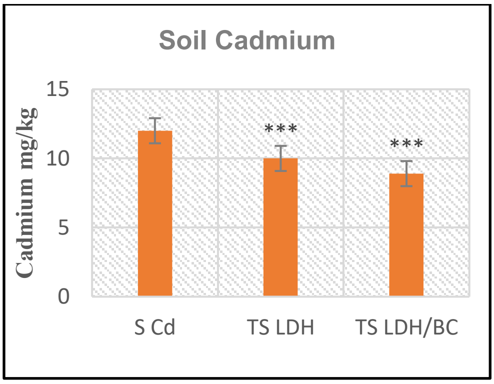

Fig. 4. Residual Soil Cadmium (mg/kg) after Treatment. (Data are presented as Mean ± SD (n=3). Asterisks indicate statistical significance compared to the cadmium-contaminated control soil using a two-sample t-test (** $p < 0.01$; *** $p < 0.001$; ns = non-significant)

The level of Cadmium in the control soil was lower than the minimum reporting level (<MRL) but the artificially added cadmium contamination for the study was 12mg/kg. Cadmium concentration decreased from 12 mg/kg to 10 mg/kg following LDH treatment and further reduced to 8.9 mg/kg after LDH/Biochar composite amendment. The results show that the Cd adsorption capacity is maximum for LDH/Biochar composite.

3.5. Soil microbial Concentration.

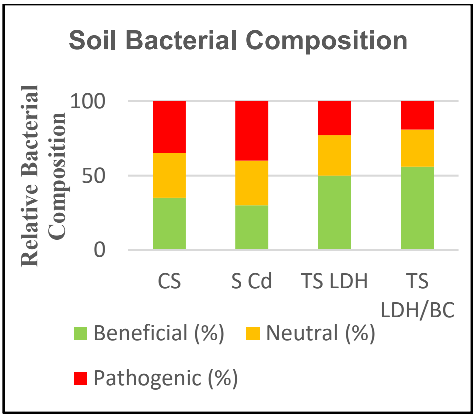

Fig. 5. Relative Bacterial Composition of control and treated soil

Microbial analysis based on culture showed apparent contrast in untreated and treated soils. The experimental sample of the untreated cadmium-contaminated soil showed uncountable bacterial growth, suggesting microbial stress and uncontrolled populations domination. Conversely, the LDH- and LDH/Biochar-treated soils exhibited countable populations of bacteria of about 10^6 CFU indicating partial restoration of the microbial activity after the treatment.

The biochemical and gram staining tests revealed that in the untreated soil there was higher presence of genera which is frequently reported as Opportunistic or pathogenic in contaminated environments, such as Salmonella and Citrobacter. However, the bacterial genera in the treated soil were commonly associated with soil nutrient recycling and environmental resilience such as Bacillus spp. and Enterococcus spp. These changes demonstrate that amendments based on LDH caused an effect on microbial community structure to a more balanced culturable population.

4. Discussion

In the current experiment, the pH in the cadmium polluted soil was of moderate acidic level, but after the amendments of LDH and LDH/Biochar, pH changed to close to neutral and even alkaline. This change was more experienced in the LDH/Biochar-treated soil, which showed high alkaline nature of the composite material. The noted rise in pH is directly connected with immobilization of cadmium, because with the rise in soil pH, soluble Cd species are converted to less bioavailable forms having reduced mobility and lower environmental risk such as cadmium hydroxides and carbonates [1,3,10]. Besides changing pH, soil organic carbon was significantly increased only in LDH/Biochar-treated soil whereas the results of LDH alone showed little variation. This trend indicates that the biochar element contributed majorly to the enrichment of carbon. The high level of organic carbon probably increased the immobilization of cadmium by means of providing oxygen-containing functional groups that have the ability to complex with the metals, thus, enhancing adsorption activities in the soil matrix [2,6,11]. There was an increment in total nitrogen content in both LDH-treated and LDH/Biochar-treated soils, but more so in the composite-amended soil. The trend shows that LDH layers enhanced the nitrogen retention mechanism via anion exchange and adsorption [4] and the porous nature of biochar reduced the nutrient loss further due to its enhanced retention in the soil aggregate [6,7,12]. Along with all the other physicochemical alterations, the level of residual cadmium concentration reduced by 16.67% with LDH and 25.8% with LDH/BC their addition . This decrease is explained by the ion-exchange and surface complexation processes inherent to LDH materials [4,5]. The high efficiency of the LDH/Biochar composite is justified by synergistic interactions between LDH layers and biochar surfaces that enhanced the amount of adsorption sites and the precipitation processes that are pH-dependent [6,7,11]. The microbial analysis revealed the microbial shift towards a more stable and nutrient recycling genera of microbes, Despite the fact that the microbial analysis was restricted to culture-based methods, the fact that the growth was uncountable in untreated soils and stable and countable in treated soils represent the indication of reduced metal stress and increased microbial tolerance. All these findings portray that not only did LDH/Biochar amendment increase cadmium immobilization but it also increased key quality indicators of the soil such as pH balance, organic carbon and nitrogen retention. The synergistic effect of the amelioration of soil chemistry and nutrient status indicates the additional benefit of the composite material compared to LDH used individually in remediation of the soil.

5. Conclusion

This experiment shows, in a controlled laboratory-based study that Layered Double Hydroxides (LDH) and LDH/Biochar mixtures can tackle the decrease of cadmium levels in the polluted soil. The LDH and LDH/Biochar addition enhanced the vital soil properties and reduced the cadmium levels in the contaminated soil substantially. Overall, the LDH/Biochar composite was more efficient in cadmium immobilization compared to LDH alone. The aforementioned increase in the carbon and nitrogen concentration in the soil also suggests that LDH/Biochar is a potential amendment to the soil and requires more research under field-scale settings.

6. Reference

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