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Research Article

Characteristics of Heavy Metal Pollution in Industrial Sludge and an Environmental–Friendly Removal Method

Nguyen Thuy Chung[1*], Luong Thi Mai Ly[2], Nguyen Xuan Binh[3], Pham Minh Chinh[4]

[1] School of Environmental Science and Technology, Hanoi University of Science and Technology; chung.nguyenthuy@hust.edu.vn
[2] Faculty of Environmental Sciences, University of Science, Vietnam National University, Hanoi; luongmaily@hus.edu.vn
[3] Institute of Science and Technology – Ministry of Public Security; binh14.163.3@gmail.com
[4] Faculty of Environmental Engineering, National University of Civil Engineering; chinhnkt16@gmail.com

***** Corresponding author: chung.nguyenthuy@hust.edu.vn; Tel.: +84–886114668

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Abstract: Sludge from wastewater treatment plants (WWTPs) in industrial park is currently a serious problem in Vietnam as well as many countries around the world. Unlike other by–products, sewage sludge from WWTPs contains a lot of toxic components, heavy metals, persistent organic substances and many other hazardous ingredients in high concentrations. Up to now, there has not been a Vietnamese study focusing on systematically assessing the level of toxic pollutants in industrial sludge in Vietnam. Therefore, this study focuses on evaluating the characteristics of industrial wastewater sludge in a specific industrial park, and thereby determining the characteristics and current status of heavy metal pollution in the sludge compared with agricultural soil samples. This study determined the heavy metals enrichments and their possible sources in industrial sludges from different sampling time. The results show that industrial sludge exhibits very high pollution for some typical heavy metals, especially Cu and Cd. The analysis of the correlation relationship between heavy metals also helps to identify the source of emission of heavy metals in the sludge sample. The PI, *Igeo* indexes are also 2–10 times higher than the control soil samples. In addition, the study also used citric acid, GLDA and ascorbic acid solutions as a method of heavy metal extraction from sludge with relatively high efficiency (~80%). Among the chelators, GLDA can be selected as the most effective removal with high capacity to remove Zn and Pb.

Keywords: Industrial sludges; Heavy metal removal; Pollution; Enrichment.

1. Introduction

Industrial sludges from various industries contain trace metals, organic compounds, macronutrients, micronutrients, organic microbial contaminants, microorganisms and eggs of parasitic organisms [1–3]. Previous studies have shown that the annual amount of sludge is constantly increasing due to urbanization and industrialization. Most of the hazardous heavy metals (Zn, Sb, Cr, Ni, Hg, Cd, Sn and V) are found in the sludge with relative concentrations many times higher than the allowable limit [1]. When arbitrarily disposing of industrial sludge into the environment, heavy metals will easily spread to surface water, groundwater and seep into the ground. Heavy metals usually exist in sludge in 5 forms: ionic

form, carbonate bound form, bound form inside or outside the solid particle mass with iron oxide and manganese, bound form with organic compounds, inert form, stable in the environment with mineral grain structure, difficult to be released under natural conditions [3].

Heavy metals such as Cr, Ni, Cu, As, Cd and Pb have been recognized as hazardous elements for the environment. The occurrence of heavy metals in the industrial wastewater and sludge are of interest because they would be often presented at considerable quantities and if leaked into surface waters or arable land, that can have severe effects on the environment and public health [4–5]. Sludge which disposes from different industries contains trace metals, organic compounds, macronutrients, micronutrients, organic micro pollutants [6]. So, the accumulation of industrial sludge poses environment problem and the bioavailable fractions of these wastes may result in secondary environmental pollution. Therefore, contamination of environment by heavy metals from untreated wastewater and sludge of various industries is a worldwide environmental problem [6–8]. Unlike organic wastes, heavy metals are non–biodegradable and thus must be treated to avoid polluting the environment. [5] studied the effect of heavy metals in sewage sludge applied to soil on its metal availability and the growth and yield of crops. Their results indicated that the yields of both cereals and legumes in dressed regions were lower than those of control regions.

In Vietnam, there are clear signs of heavy metal pollution from industrial sludges. The industrial waste problem has become one of the prime concerns in many provinces of Vietnam. Many industries have set up in and around the cities during the last decade, and the number of new industries is continually increasing. In recent times, the rapid development of various industries has created environmental problems that pose a serious threat to the environment [9–12]. A recent study shows that although environmental management has improved in recent years, heavy metal pollution levels are still high in sludge from Hanoi's Kim Nguu River. Most of the sludge samples here have concentrations of Cr, Ni, Cu, As, Cd and Pb exceeding the permissible standards of Vietnam (QCVN 50: 2013/BTNMT), which is caused by industrial wastewater discharged into the Kim Nguu River [12]. This result is in stark contrast to the situation in the Mekong Delta, which is less polluted by industrial activities [10]. The risk of environmental pollution from sludge can be found in Vietnamese statistical reports around Ho Chi Minh City [13–14] where there are many industrial plants but information on composition and volume sludge is still a gap that does not meet the current environmental management needs of Vietnam.

Various surveys of the heavy metal concentrations in sewage sludge have been undertaken to evaluate the suitability of sludge for land application. It is particularly important to study ecological risk assessment to industrial sludges because modern industrial areas are often densely populated due to the presence of industrial and commercial activities as well as easy access to amenities such as transportation, electricity, water, entertainment, and healthcare.

Therefore, this paper studies the characteristics of sludge in an industrial park in the Northern province, thereby assessing the pollution level of heavy metals in the sludge samples. In addition, the study also identifies the emission sources of toxic substances in the analyzed sludge sample, find an appropriate method of recovering heavy metals from the sludges. This study aims to propose an efficient and friendly method of heavy metal extraction from the sludges.

2. Materials and Methods

2.1. Collecting sludge samples

Sludge samples were collected from wastewater treatment plants located in an industrial park (BT, 11 samples) and agricultural soil (control, 4 samples) in the North of Vietnam.

Industrial sludges of the industrial park were collected and managed in the form of ordinary solid waste. Each factory that generates heavy metals in conventional sludge has its own wastewater treatment system, treating heavy metals and some substances before being poured into the general treatment system of the industrial park. However, industrial sludge is generated during wastewater treatment. Industrial wastewater, which is treated in wastewater treatment plants, usually meets column B standards according to QCVN 40:2011/BTNMT before being connected to a common wastewater treatment plant of industrial parks [15]. The industrial park's wastewater treatment plant has been synchronously invested and built by a leading unit in the field of wastewater treatment plant construction for industrial zones. The wastewater treatment system met the standard of grade A wastewater treatment (QCVN 40:2011/BTNMT) with the total capacity according to the design of 4 modules is 10,000 m^3/day. The water collection system in the industrial parks is MMBR system (Moving bed biofilm reactor system) and they had the standard for centralized WWTP. Currently 1 module has been operating with a capacity of 2,500m^3/day. The wastewater treatment system has been fully trained and transferred to the industrial park for operation. Industrial wastewater is treated at each wastewater treatment plant, after partially resolved the heavy metal component, it is brought into the centralized water treatment area. The industrial park which was chosen in this study has 23 manufacturing companies, including 7 production facilities with wastewater containing high heavy metal content, including metallurgical, mechanical and chemical plants.

2.2. Sample pre–treatment

Sludge samples were taken 4 times in a year 2020, total samples were 12, each sample weight was 200 grams and stored in sealed zipper bags. In the sludge storage area, a small shovel was used to scoop up samples at 5 points, then mix well and take 200 gram each. Some indicators such as pH, EC, ORP, COD, T–N, T–P are analyzed at the wastewater treatment station [16]. After collection, the samples were stored in sealed foam containers, in a cool place. Samples were moved to the laboratory according to TCVN 6663–15:2008 (ISO 5667–15:1999). The collected sludge sample is dried in a dark and closed room, then the sample is crushed, removed impurities, sieved through a sieve with a pore size of 0.63 µm and collected samples with a particle size < 0.63 µm to analyze the metal content in the most active sedimentary phase, containing mainly clay and meat particles. Samples were stored in a deep refrigerator waiting for analysis, before analysis the samples were left at room temperature and the drying coefficient was determined according to TCVN 4080:2011. Industrial sludge is collected at the mud drying yard by suitable tools. For the control soil sample, 4 agricultural soil samples were also taken for comparison as the background value for each sampling time. We aim to compare the industrial sludges with agricultural soil samples for risk assessment. The soils is nearby the industrial park and can be shown that they were not contaminated. The morphology and elemental contents on the surface of the investigated sludge samples were observed and analyzed using a scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) techniques (SEM–EDS, JEOL JSM–7600F using its variable pressure mode and an accelerating voltage of 15 kV).

2.3. Total metal concentration analysis

Total heavy metal analysis was performed in accordance with the sample handling procedure for the analysis of Cd, Cr, Cu, Pb, Ni and Zn metals, which was conducted according to the guidelines of EPA 3050B [17]. Analytical grade (AG) chemicals, procured from E–Merck, India, were used throughout the study without any further purification. The metal standards were prepared from stock certified standard solution of 1000 mg/l (Merck, Germany) by successive dilution with ultra–pure water (TKA Milli–Q Ultra–Pure Water System, Germany). The analytical quality control was assured by standard operating

procedures, repeated analysis of reagent blanks and recovery analysis of several spiked samples. Other parameters were analysed using the standard methods of US. EPA.

2.4. Assessment of heavy metal pollution according to pollution indicators

2.4.1. Pollution Index (PI)

PI: Single pollution index of heavy metals, determined according to the following formula:

$$\mathrm{PI} = \mathrm{TE(industrial\ sludge\ sample)/TE(control\ sample)} \quad (1)$$

where TE (waste sludge) is the average value of heavy metals in the sludge; TE (control soil) is the mean value in the baseline soil sample; PI is classified as follows: low (PI ≤ 1), moderate (1 < PI < 3), high (PI ≥ 3) [13].

2.4.2. Geoaccumulation indexes (Igeo)

Igeo assesses contamination by comparing the total metal content of the sample with the background value of that metal [14].

$$\mathrm{Igeo} = \log 2 \frac{\mathrm{Cn}}{1{,}5\mathrm{Bn}} \quad (2)$$

where Cn: Metal content in the sample; Bn: Base value of metals in the Earth's crust; 1.5: The factor is given to minimize the impact of possible changes to the background value due to lithological changes in the sediment.

where Igeo ≤ 0: Not contaminated, 0 ≤ Igeo ≤ 1: not– average contaminated, 1 ≤ Igeo ≤ 2: average, 2 ≤ Igeo ≤ 3: average– heavily contaminated, 3 ≤ Igeo ≤ 4: heavily contaminated, 4 ≤ Igeo ≤ 5: heavily and seriously contaminated, 5 ≥ Igeo: seriously contaminated.

2.4.3. Treatment of heavy metals in sludge using environmentally friendly chemicals

In this study, two types of chelator solutions were used: 1. Solution of N, N–Dicarboxymethyl glutamic acid tetrasodium salt (GLDA), ascorbic acid and citric acid. The concentrations of the single washing agent are 200 mM for all chelators. The initial solution of heavy metals (3 metals: Pb, Zn, Cu, with concentration corresponding: 300, 1200 and 150 mg/L) were used to investigate the removal effects on heavy metal washing efficiency. The concentrations were similar with heavy metal concentration in the types of industrial sludges. The reaction time was 24 hours with pH 7.0. A solution of 0.1 N HCl were used as control solution in all experiments. pH values of the washing solution were adjusted using HCl and NaOH. This study was similar with method [5].

2.4.4. Statistical data processing

The classical statistical analyzes were processed using IBM SPSS software version 20. The probability level P < 0.05 was considered to be significant.

3. Results

3.1. Sludge characteristic

After processing and collecting data, a table of hysicochemical properties and SEM images of the sewage sludge samples of the research subjects were presented as follows (Table 1). 4 samples of industrial sludges among 12 samples were chosen to investigate the characteristic. According to QCVN 50:2013/BTNMT, the pH of sludge with pH ≥ 12.5 or pH ≤ 2.0 is defined as hazardous sludge. Looking at the data table, it can be seen that the pH

of the sludge sample ranges from 6.05 ± 0.1 to 6.07 ± 0.12, all of which are neutral for industrial sludge. Total organic carbon fluctuated 634 ± 40.88 mg/L.

Table 1. Summary of sludge characteristic.

Parameters	Sample 1	Sample 2	Sample 3	Sample 4
pH	6.3	6.5	6.4	6.3
ORP (mV)	230	350	402	389
COD (mg/L)	1203	1504	1315	1420
TOC (mg/L)	728	837	204	736
T–N (mg/L)	612.3	936.6	699.4	777.5
T–P (mg/L)	45.1	23.1	32.0	34
Potassium (K)	203	201	210	190
Magnesium (Mg)	102	120	115	113

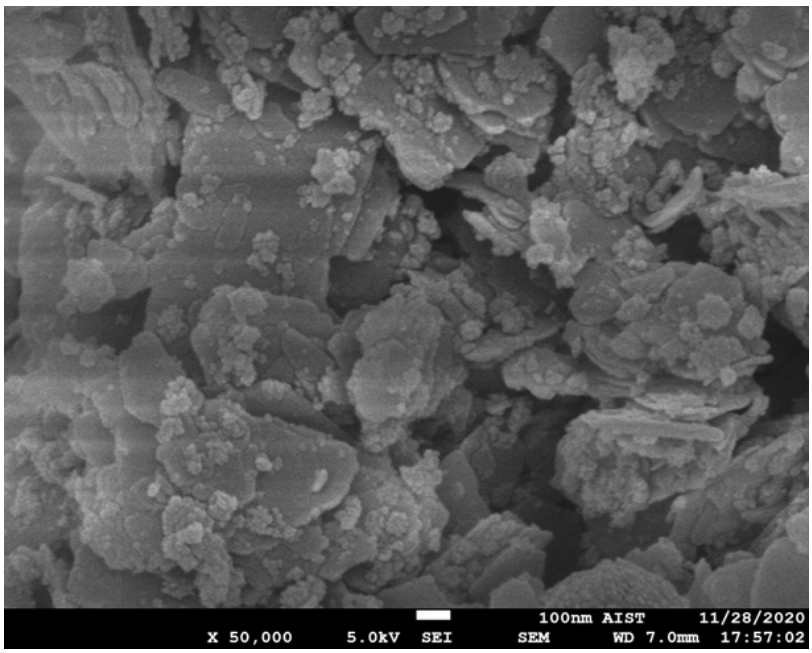


Figure 1. A scanning electron microscope (SEM) of the sludge.

Figure 1 shows microscopic images of representative samples of sludge from the industrial site. In general, the surface morphological characteristics of the sludge are relatively uniform. The high proportions of elemental oxygen and carbon in the sludge samples indicate a large amount of organic matter in the sample, possibly as a result of coagulation of the polymers during the treatment process. The similarity in surface morphology of these samples also supports this conclusion.

3.2. Heavy metal concentrations

The average concentrations of Cd, Cu, Pb, Zn in the sludge samples of the industrial park had the average concentrations of the elements 0.9 mg/kg, 297.2 mg/kg, 164.6 mg/kg and 1177.4 mg/kg, respectively. Most of the elements analyzed were lower than the maximum allowable concentrations for normal sludge (QCVN 50: 2013) (10 mg/kg; 300 mg/kg; 5000 mg/kg, respectively). In some cases, some sludge samples from Ba Thien Industrial Park exceeded the allowable limit for Pb. Therefore, these sludges are considered as hazardous solid wastes, which are not treated and buried according to regulations [15].

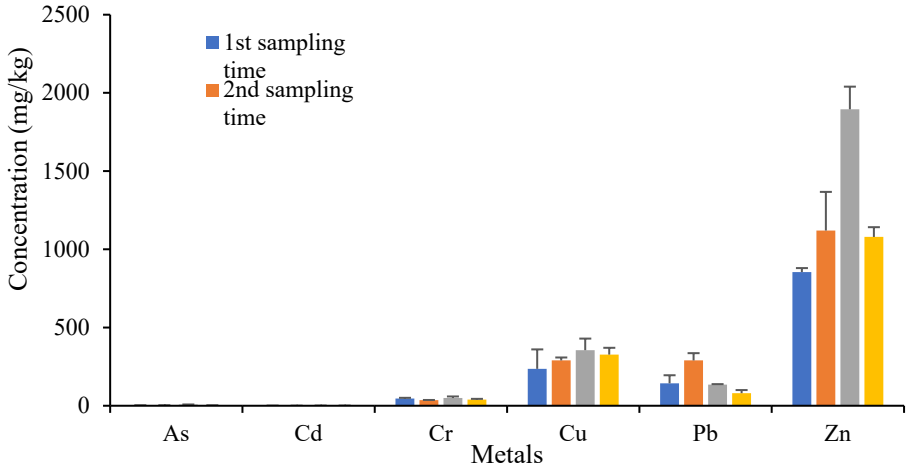


Figure 2. Heavy metal total concentrations in sludges.

3.3. Pollution Index calculation in the industrial sludges

Table 2 describes the pollution index of industrial sludges. The PI (the ratio of TE concentration in sludges to that in soils) was > 3 for Zn, Cu, V and Cr in all samples (Figure 3). Furthermore, the medians of the values were > 5, indicating that these heavy metals were highly enriched in the sludges according to the classification [18].

Table 2. Pollution index of industrial sludges.

	Fe	V	Cd	Cr	Cu	Ni	Mn	Pb	Zn
PI1	64.22	8.54	5.06	9.41	19.87	7.91	3.56	1.65	23.88
PI 2	60.09	11.25	5.52	13.78	35.06	5.12	5.34	1.74	19.83
PI3	95.87	13.96	5.06	16.47	33.26	7.44	6.88	3.97	19.17
PI4	80.28	17.50	4.14	15.13	6.56	3.95	9.61	2.81	17.07
PI5	81.19	19.38	3.22	12.10	8.54	5.12	6.77	2.08	15.38
PI6	130.73	17.50	5.52	19.16	48.54	11.63	5.22	8.24	17.32
PI7	95.87	9.79	4.14	12.77	22.47	5.12	3.68	5.50	14.56
PI8	83.94	20.83	2.76	12.10	26.07	5.58	6.77	11.30	17.28
PI9	87.16	11.88	2.76	11.09	29.66	6.98	7.00	9.77	33.40
PI10	115.14	27.08	3.68	21.51	41.35	10.23	9.97	3.97	40.78
PI11	96.79	12.71	5.52	11.76	22.47	2.51	7.00	4.27	32.82

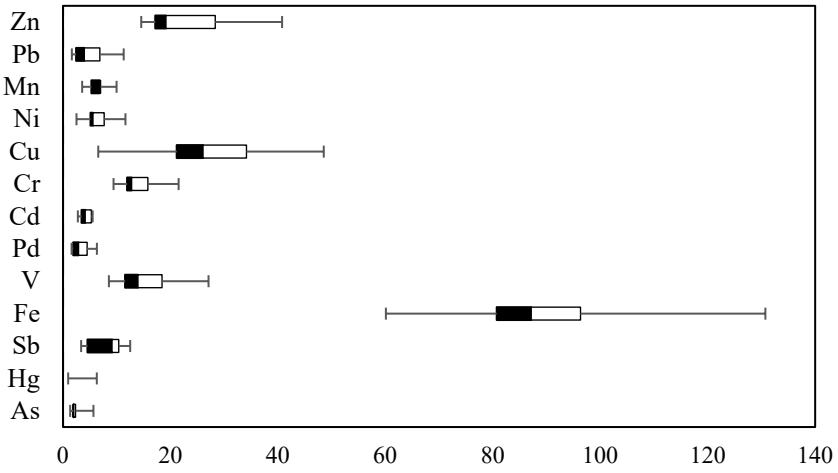


Figure 3. Comparation between industrial sludges and control samples.

In general, compared with the natural soil control sample, most of the PI of metals in the wastewater samples from the industrial park are high. In which, Fe is the metal with the highest PI index, at a high level (PI ≥ 3). Pb has the lowest PI, which is low (PI ≤ 1).

Compared with the control sample of sewage sludge from the groundwater treatment plant, most metals have medium PI, Cd is the metal with the highest PI index, at high level (PI ≥ 3). Fe is the metal with the lowest PI, all PI values < 1, at a low level (PI ≤ 1). This shows that the metal concentration distribution for the ground soil and sludge samples is not the same. For the sludge samples, the metal concentration is distributed more evenly and averagely than in the natural soil environment. Similar to industrial park sludge samples, Fe and Zn are two components that have great influence on the natural soil environment [2, 19, 20].

3.4. Geoaccumulation indexes (Igeo)

There are different indexes generally used to identify metal concentrations of environmental concern like: the metal enrichment factor (EF) and geoaccumulation indexes (Igeo) [8–9]. These indexes identify, numerically, pollution level soils and normally they are calculated on the soil exchangeable fraction because it represents the real bioavailable fraction. The bioavailable metal content in soil exerts a decisive impact on soil quality and it's used in food production. Hence, the assessment of metal contamination is of vital importance in farming areas.

Based on the data in the Table 3, the pollution level based on the Igeo index, it can be seen that: In general, most metals have medium–high Igeo values. In which, Fe has the highest average Igeo value, or in other words, the highest level of Fe contamination. Hg is the metal with the lowest mean Igeo value (all values ≤ 0).

Table 3. Igeo index in industrial park sludge.

Metals / Name	As	Sb	Fe	V	Cd	Cr	Cu	Ni	Mn	Pb	Zn
BT1	–0.17	1.42	5.42	2.51	1.87	2.64	3.73	2.36	1.25	0.13	3.99
BT2	0.37	0.68	12.51	4.58	–0.91	4.19	7.44	2.29	7.64	4.66	8.83
BT3	0.60	1.70	13.18	4.90	–1.03	4.44	7.36	2.83	8.01	5.85	8.78
BT4	0.15	0.26	12.93	5.22	–1.32	4.32	5.02	1.92	8.49	5.35	8.61
BT5	0.42	0.72	12.94	5.37	–1.68	4.00	5.40	2.29	7.98	4.92	8.46
BT6	0.46	1.90	13.63	5.22	–0.91	4.66	7.91	3.47	7.61	6.91	8.63
BT7	0.68	2.09	13.18	4.38	–1.32	4.08	6.80	2.29	7.11	6.32	8.38
BT8	0.37	2.15	12.99	5.47	–1.91	4.00	7.01	2.42	7.98	7.36	8.63
BT9	0.42	1.53	13.04	4.66	–1.91	3.87	7.20	2.74	8.03	7.15	9.58
BT10	1.76	1.77	13.45	5.85	–1.49	4.83	7.68	3.29	8.54	5.85	9.87
BT11	1.92	1.87	13.20	4.76	–0.91	3.96	6.80	1.26	8.03	5.96	9.55

3.5. Heavy metal recovery experiment

In this study, sewage sludge was used as the subject of metal recovery study. Chemical analysis using the ICP–MS method showed that the sludge was heavily contaminated by heavy metals, especially zinc (Zn) with concentrations higher than 1200 mg/kg. Therefore, the study used 3 types of specific chelators to test the ability to recover 3 typical heavy metals in wastewater.

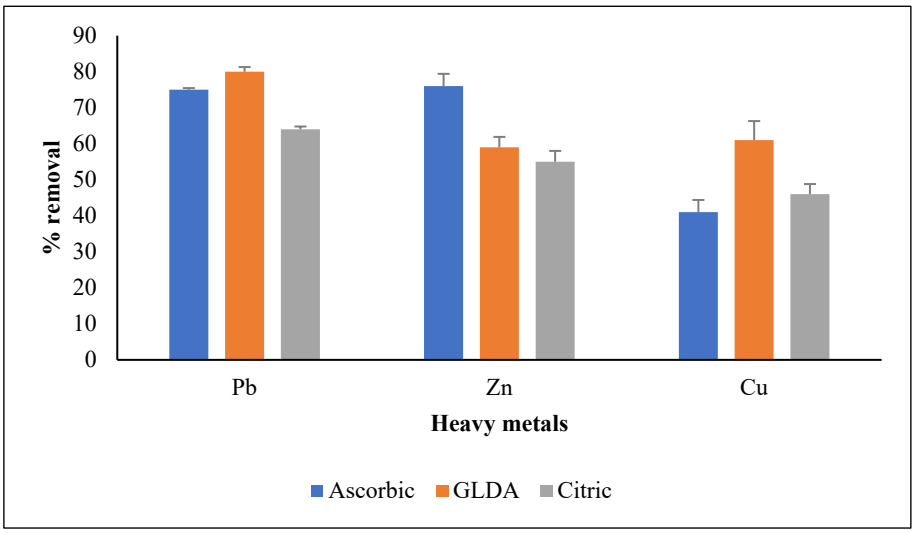


Figure 4. Effect of different extractant concentrations on metal removal.

Overall, among the studied chelators, ascorbic acid was the most effective in extracting Zn from contaminated sewage sludge, followed by GLDA and citric acid (Figure 4). GLDA performed best in extracting Pb and Cu from contaminated sludge although the overall removal efficiency was not really higher than the other 2 chelators for Pb. It can be seen that citric acid has the worst removal efficiency compared with other chelators under the same study conditions.

More importantly, 80.1% Pb, 76,5% Cu, two metals with levels exceeding the national allowable limit were extracted from the sludge using only 200 mM recoverable ascorbic heavy metal. The results of the study open up new application directions for environmentally friendly chemicals used to replace strong acids in cleaning and recovering heavy metals in polluted sludge.

4. Discussion

Waste sludge at Ba Thien 2– Vinh Phuc Industrial Park and industrial sludge collected at Thanh Cong 2 Cement Plant both show very high pollution for some typical heavy metals, especially Cu and Cd. Among the heavy metals, Cadmium has the highest ecological potential risk compared to other elements.

Sludge from the two sampling points above is considered hazardous waste. The results of analysis of pollution index (PI) and ecological risk index (RI) both show that the sludge sample has 2–10 times higher results than the control sample. Industrial sludges have a higher immobilization capacity, with (–COOH) and (–OH) being the typical functional groups present on the sludge surface.

The Pollution Index (PI) in metals and Geoaccumulation Index (Igeo) are indicators studied to calculate the presence and intensity of anthropogenic contaminant deposition on soil or sludges. These indexes of potential contamination are calculated by the normalization of one metal concentration in the research sample respect to the concentration of a reference element. In this study, although the geological accumulation risk indicators are at a moderate level of pollution, the ecological risks of each metal Er and the pollution index of each metal are quite high, potentially causing polluted environment. Therefore, it is necessary to strictly manage as well as take reasonable measures to handle and avoid risks.

The final result shows that most heavy metals in the sludges samples were lower than the maximum allowable concentrations in standard guideline (QCVN 50: 2013) (10 mg/kg; 300 mg/kg; 5000 mg/kg, respectively). But specifically, lead, zinc and copper in some sludge

samples exceeding the national allowable limit so these metals were chosen for removal study. Lead and copper were extracted from the sludge using only 200 mM recoverable ascorbic, GLDA and citric solution that worked effectively. The results of the study open up new application directions for environmentally friendly chemicals used to replace strong acids in cleaning and recovering heavy metals in contaminated industrial sludges.

5. Conclusions

It is essential to determine the heavy metal concentrations in the industrial sludges to select appropriate disposal methods. We conducted a survey of heavy metal concentrations of sludge samples from 11 industrial sludge samples from an industrial park located in Vinh Phuc Province. The average concentrations of Cd, Cu, Pb, Zn in the sludge samples of the industrial park had the average concentrations of the elements 0.9 mg/kg, 297.2 mg/kg, 164.6 mg/kg and 1177.4 mg/kg, respectively. This study also characterizes the physico–chemical characteristic of industrial sludge. Environmental issues related to possible management options are also addressed. Sludge samples from industrial parks were analysed and calculated the PI and Igeo index in comparison with the natural soils. The results indicate that pollution indexes of Cu, Pb, and Zn could be 2–10 times higher than the control natural soil and it may pose a potential threat to the water quality for sludge dumped near water bodies. Therefore, we recommend avoiding uncontrolled upland disposal of such sludge. Some preliminary treatment to remove heavy metals from the sludges should be applied. A recovery of Cu and Pb using GLDA solution from this sludge could be considered with high efficiency.

Supplementary Materials: None

Author Contributions: Conceptualization, N.T.C., L.T.M.L.; methodology, N.X.B.; software, P.M.C.; validation, N.T.C., L.T.M.L., N.X.B.; formal analysis, P.M.C., N.X.B.; investigation, L.T.M.L.; data curation, N.T.C.; writing–original draft preparation, N.T.C., P.M.C.; visualization, L.T.M.L.; project administration, N.T.C.; All authors have read and agreed to the published version of the manuscript".

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