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Foreword

Welcome to ESTEEM Volume 2. In this issue, we address a gamut of topics from the engineering disciplines to language education. We hope that ESTEEM, by publishing articles from a diverse range of disciplines, will encourage debate and exchange among researchers from assorted academic backgrounds.

I would like to thank our advisor, Prof. Madya Mohd Zaki Abdullah for his distinctive imprint on this edition. His leadership of the journal in its 2nd year of growing impact and reputation has been outstanding. His vision, commitment to excellence, and attention to detail are widely recognized by the Penang academic community as determining factors in the journal's success so far. We will do our best to continue and expand on this tradition of excellence.

Since its launch in 2003, ESTEEM is indeed fortunate to have a dynamic Editorial Team. These people have provided the journal with an outstanding service of reviewing submissions for publications. The journal follows the established policy of a blind review process consisting of at least two peer reviewers per submission. We depend upon their knowledge and judgement in advancing the scope and utility of this journal. Without their support and enthusiasm none of this would have been possible. Also, my thanks to all the contributors, both the successful and not so successful.

Our vision of the ***ESTEEM*** journal is that it should be the journal that belongs to you, the academic and research community. This includes all engineers and academicians working to unravel the mysteries of research, teaching and learning, in all its facets. We wish the journal to be responsive to your needs and your interests. Please feel free to contact any of us in the editorial board to give us your ideas and suggestions for the development of the journal. We look forward to working with you all in expanding this emerging venue for communicating high quality research on the many aspects of academia.

Finally, I would like to take this opportunity to invite all authors and readers to contact me at **esteem@ppinang.uitm.edu.my** to share their comments and advice on how to further enhance the journal's value to the wider research community in knowledge and how to move ESTEEM to the next level of excellence.

The Chief Editor
May, 2005

A Comparative Study between Cu and Pb in the Speciation Study of Heavy Metals in a Stabilization Pond Treating Household Wastewater

Lim Boon Tik

The waste stabilization pond system in Taman Kota Permai, Seberang Perai Tengah, Penang consisting of two facultative ponds in parallel and a maturation pond connected to each of them in series is being used to treat domestic sewage. This study investigates the removal efficiency of trace metals (Cu and Pb) and their speciation in the particulate of this stabilization pond system.

The percentages of removal efficiencies of trace particulate heavy metals along the treatment path were 62% for Cu and 65% for Pb. The speciation of particulate heavy metals was carried out according to the procedure which partitions particulate trace metals into five fractions, namely, (a) exchangeable, (b) bound to carbonates, (c) bound to Fe-Mn oxides, (d) bound to organic matter and (e) residual. The results showed that Pb appeared mainly in the bound to Fe-Mn oxides fraction. The percentage distribution of Pb in this fraction in the particulates of the final effluents was 69 %. However, Cu was mainly found in the bound to organic matter fraction and its percentage distribution was 64%. The results also shows that Pb is more mobile than Cu. The percentages associated with mobile phase is 80% for Pb while the mobile phase for Cu is 34% only.

Introduction

Stabilization ponds have been used for sewage treatment in places where land is easily available and temperature is most favorable for their operation. In Malaysia, the ponds system is popularly used to treat palm oil mill effluent, rubber factory waste and domestic waste to reduce the BOD and suspended particles prior to their discharge into watercourses. Evaluation of the performance of this type of treatment system by

Asairinachan (1979) and John (1984) generally shows that it is effective in reducing, among others, the BOD and suspended solids in the effluents. However, very few information regarding the efficiency of the removal of heavy metals in this system have been reported. Kaplan *et al.* (1987) studied a waste stabilization pond system treating domestic wastewater and found that a decrease in the total concentrations of Zn, Cu, Pb and Cd was attained during the various stages of treatment.

However, the percentages of heavy metals in the dissolved fraction were found to increase along the treatment path. Sewage treatment ponds may receive effluents from both domestic and industrial sources, as well as intermittent influxes of storm water runoff from nearby areas. All these sources produce effluents contaminated with heavy metals and some portions of these metals will pass through the treatment process to be discharged to surface waters (Lester *et al.*, 1979). If there is a trend towards the greater use of sewage treatment ponds to treat industrial effluents, a better understanding of the role of treatment processes in metals abatement (Brown & Lester, 1979) is warranted. The efficiency with which a sewage treatment pond system retains influent metals will depend upon the physico-chemical forms of the metals and their response to the physical and biological treatment processes. Therefore, knowledge of the metal speciation may go a long way in understanding the variability in the treatment efficiencies.

The objectives of this study are:

a. to determine the removal efficiency of particulate heavy metals (Cu and Pb) in a stabilization pond system treating domestic wastewater.
b. to study the speciation of the particulate heavy metals (Cu and Pb) along the treatment path.
c. to compare the remobility between Cu and Pb in a stabilization pond system.

Material and Methods

Reagents

All Reagents Used in the Experiments are AR Grade

Experimental

Sampling

The Stabilization Pond System

The stabilization pond system for this study is located in Taman Kota Permai, Seberang Perai Tengah, Penang, Malaysia. It consists of two facultative ponds operating in parallel and a maturation pond connected to each of them in series (Fig.1). Domestic waste from the neighboring residential areas is channeled into the ponds for treatment. The effluent from the maturation pond is discharged through two outlets into the Juru River. The hydraulic retention time of the two facultative ponds was estimated to be 46 days whereas that of the maturation pond was 11 days.

Table 1: Mean Total Concentration of the Particulate Metals (μg/g) at Various Stages of Treatment

Metal	Pb	Cu
Influent	107.2 ± 9.2	940.5 ± 87.98
Facultative Pond's effluent	62.6 ± 7.8 (42%)	600.8 ± 66.83 (36%)
Maturation Pond's effluent	44.5 ± 7.1 (58%)	298.87 ± (68%)

* Numbers in parentheses indicate percentages of removal

Collection of Wastewater Samples

Wastewater samples were collected in pretreated 4 L polyethylene bottles from sampling points 1, 2, 3, 4 and 5 (Fig.1) with 1 and 2 representing inlets (influent) to the respective facultative pond, 3 and 4 the outlets (facultative) of the respective facultative pond to the maturation pond and 5 are outlets (effluent) of the maturation pond leading to the nearby river. The samples were first centrifuged at 8000 rpm for 20 minutes and the supernatants filtered through 0.45 um membrane filters to be separated into particulate and dissolved fractions. The particulate fraction of each sample consisting of the pellet and the residue was dried in an oven at 80°C for a period of a week.

Determination of Total Concentration of Eeach Metal in the Particulate

The respective dried sample (Pb & Cu) were weighted accurately and poured into each individual Teflon container. To each individual container, 5 mL 30% concentrated H_2O_2 was added, followed by 5 mL 65% concentrated HNO_3 (AR grade). Then the mixed solution was digested in Microwave Oven (Milestone Model 1200 Mega) for 25 minutes. After cooling to room temperature, the sample volume was made up to 10 mL with de-ionized water. Filtered the sample if necessary.

Total concentration of each metal was determined by using Graphite Furnace Atomic Absorption Spectrophotometer(GFAAS).

Speciation of Particulate Pb and Cu

The speciation of particulate Pb and Cu was carried out according to the sequential extraction proposed by Tessier et al. (1979). This analytical procedure was developed for the partitioning of particulate trace into the following 5 fraction: exchangeable, bound to carbonates, bound to Fe-Mn oxides, bound to organic matter, and the residual. The extractions were carried out in an acid-washed 50 mL polyethylene centrifuge tube with a screw cap. Each successive extraction was then separated by centrifugation at 3000 rpm for 30 min. The supernatant was removed with a pipette and make up to 10 mL with deionized water. All final extracts were stored in acid prewashed bottles for Pb-Cu determinations. The procedures for sequential extractions are as follows:

Fraction 1. Exchangeable: 0.2 g of dried particulate was extracted at room temperature for 1 h with ammonium acetate solution at pH 7 at constant agitation.

Fraction 2. Bound to carbonates: The residue from fraction 1 was leached at room temperature with 8 mL of sodium acetate adjusted to pH 5 with acetic acid. The solution was agitated continuously at room temperature for 5 h.

Fraction 3. Bound to Fe-Mn oxides. The residue from fraction 2 was extracted with 8 mL of 0.25 M hydroxyammonium chloride in 25 % (v/v) of acetic acid. The mixture was then heated to 96°C in a water bath with occasional agitation for 6 h.

Fraction 4. Bound to organic matter. 1.5 mL of 0.02M nitric acid was added to the residue from fraction 3 and the mixture was heated to 85°C for 2 h with occasional agitation. Then, a

second 1.5 mL aliquot of 30% hydrogen peroxide, adjusted to pH 2 with concentrated nitric acid, was then added and the sample again was heated to 85°C for 3 h with constant agitation from time to time. The solution was then cooled down to room temperature, 2.5 mL 3.2 M ammonium acetate in 20% (v/v) nitric acid was added and the sample was agitated continuously for 30 min. The addition of ammonium acetate was to prevent readsorption of extracted metals onto the oxidized particulate.

Fraction 5. Residual. The residual from fraction 4 was digested with 5 mL of concentrated nitric acid in a microwave oven model Milestone 1200.

Quality Assurance

The following were steps taken to ensure quality data:

1. All preparatory works were conducted in class 100 laminar flow clean bench.
2. Deionized water of resistivity 18 Ω - cm was used to prepare all the solutions.
3. All chemicals used were of analytical grade (AR) or better.
4. All glass wares and sampling bottles were soaked in 10% nitric acid (v/v) bath for at least 24 h.
5. Blank was used in all analysis.

Results and Dicussion

Total Metals Concentrations

The mean total concentrations of Cu and Pb as well as the percentages of removal of the two metals during the course of treatment are presented in Table 1. The data showed that the total mean concentration of Cu and Pb decreased along the treatment path, that is, from the pump house to the facultative ponds, and finally to the maturation ponds before it is discharged into Juru River. It is observed that 42% of Pb was removed in the facultative ponds while the percentages of removal for Cu is 36%. Overall, the stabilization pond system in Kota Permai was able to achieve removal effiencies of 58, and 68% for Pb and Cu, respectively. The decrease in the percentages could be brought about by the deposition of

heavy metals in the sediment or being taken by the biota like algae or fish in the stabilization ponds system.

Speciation Studies

The concentration of Pb and Cu in various fraction of the particulates are given in Table 2 and 3 respectively. Figure 2 and 3 display the speciation patterns of Cu and Pb respectively.

Table 2: Concentration and Percentage of Pb in Various Fractions of the Particulate Along the Treatment Path

	Exchang--eable	%	Bound to carbonate	%	Bound to reducible phase	%	Bound to organic matter	%	Residual	%	Total
Influent	14 ± 2.7	11	4.4 ± 1.1	4.7	83 ± 7.2	67	19 ± 2.5	16	3.2 ± 0.94	2.5	124 ± 13
Facultative pond effluent	4.8 ± 1.0	6.9	3.9 ± 1.1	5.6	48 ± 3.4	68	11 ± 1.3	16	2.2 ± 0.68	3.1	70 ± 6.3
Final effluent	1.8 ± 0.3	4.1	2.9 ± 0.6	6.5	30 ± 1.4	69	7.1 ± 1.0	16	1.7 ± 0.40	3.8	44 ± 2.8

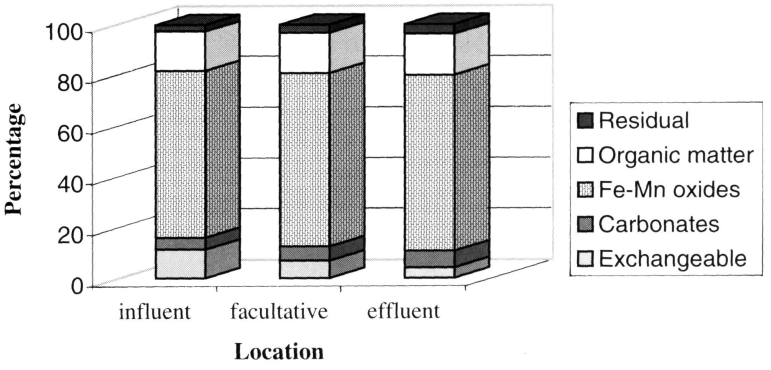

Figure 2: Speciation of Particulate Pb

Table 3: Concentration and Percentage of Cu in Various Fractions of the Particulate Along the Treatment Path

	Exchang--eable	%	Bound to carbonate	%	Bound to reducible phase	%	Bound to organic matter	%	Residual	%	Total
Influent	87 ± 17	9.9	42 ± 8.7	4.0	201 ± 39	22	567 ± 110	62	22 ± 6.5	2.3	919 ± 177
Facultative pond effluent	43 ± 9.1	6.6	33 ± 7.6	5.2	48 ± 35	23	403 ± 83	63	22 ± 6.5	2.6	647 ± 135
Final effluent	16 ± 4.3	4.6	19 ± 4.7	5.5	83 ± 20	24	224 ± 57	64	9.8 ± 3.2	2.7	352 ± 88

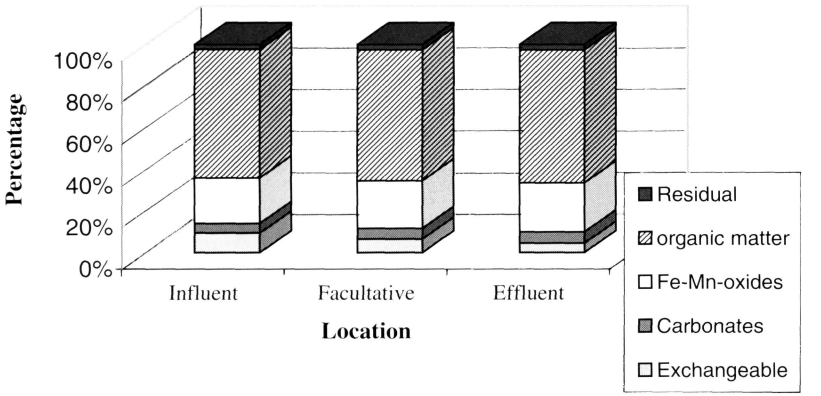

Figure 2: Speciation of Particulate Cu

Pb

Figure 2 shows that at various stages of treatment, Pb was found mainly in the Fe-Mn oxides fraction with 67, 68, and 69% for the particulates in the influent, facultative ponds effluent and final effluent, respectively. In contrast, only 16% of Pb was found at each stage for organic matter fraction. Table 2 showed that the percentage of Pb in the exchangeable fraction of the raw influent, facultative ponds effluent and final effluent was 11.3, 6.9 and 4.1%, respectively. As heavy metals in this fraction are easily removed from the particulate, this shows that Pb in the raw influent was more easily remobilized than when it was in the facultative effluent or final effluent. In other words, the mobility of Pb reduced as it moved along the treatment path. In contrast to the exchangeable fraction,

the percentage of Pb in the bound to carbonate fraction increased along the treatment path. The pattern of distribution of Pb in the residual was similar to that found in the bound to carbonate fraction, its percentage of distribution also increased along the treatment path. The low percentage of Pb found in the residual fraction indicates that the metal was mainly brought about by human activities.

Copper

By referring to Figure 3, the most remarkable feature of copper is its close association with the organic matter faction. The percentages of copper attached to the organic matter at various stages of treatment were 61.66, 62.77 and 63.7%, respectively. This trend is typical of Cu in the sediments or suspended particulates (Solomons and Forstner, Izquierdo C. *et al.*,1997; Zuflaurre *et al.*, 1998) and waters (Smines, 1983). The speciation pattern of Cu indicated that more than 61.66% of the metal was associated with organic matter at various stages of treatment. The percentages of Cu attached to the Fe-Mn oxides along the treatment path were 22, 23 and 24% respectively, and these were much lower than Pb. Similarly, the percentages of Cu in the residual fraction was very low (less than 2.33% at all stages), this indicated the presence of Cu in the stabilization ponds system was brought by human activities. The percentages of Cu attached to the bound to carbonates fraction along the treatment path are 4.6, 5.18 and 5.47%, respectively. The pattern of speciation of Cu for this fraction is similar to that of Pb, that is, its concentration increased along the treatment path.

Remobilization Capacities of Pb and Cu

The study of the speciation of heavy metals in different fraction can lead to the determination of their contamination risks (Forstner, 1985; Pardo et al.,1990). The more mobile or more dangerous phases in the speciation scheme are exchangeable, bound to carbonate and bound to Fe-Mn fractions. The distribution Pb and Cu in the mobile phase was presented in Figure 4. The results showed that Pb was more mobile and its percentages associated with the mobile phase in the final influent is 80%. In other words, Pb had the potential of being released to the environment if there were changes in the pH, salinity and redox potentials or the presence of natural and synthetic chelators (Forstner and Wittmann, 1983) However, Cu was observed to be less mobile as compared to Pb and its percentage associated with the mobile phase was only 34% (Laxen. 1981).

Table 4: Percentages of Mobile Pb and Cu in the Particulate Along the Treatment Path

Types of Metal	Pb	Cu
Influent	83.0%	36.1%
Facultative Pond effluent	80.7%	34.7%
Final effluent	79.9%	33.7%

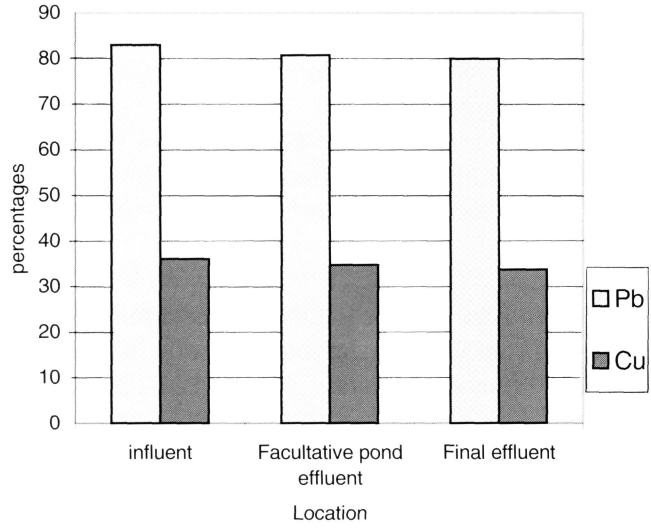

Figure 2: Comparison of Mobile Fraction Between Particulate Cu and Pb at Various Stages of Treatment

Conclusions

Based on the discussion above, it is apparent that the stabilization pond system studied could remove considerable amount of particulate Pb and Cu in the wastewaters. Pb was found to be more mobile as compared to Cu. Pb appeared mainly in the Fe-Mn oxides fraction while copper was mainly associated with the organic matter fraction.

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