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Performance of Oxidation Ponds in Removing Heavy Metals from Pig Farm Wastewater

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Abstract: Pig manure is a source of heavy metals which may cause pollution if not properly managed. Oxidation pond system is an economical method of wastewater treatment commonly used in pig farms to reduce water pollution. However, the fate of heavy metals in different oxidation pond systems in pig farm are lacking in literature. Therefore, in this study heavy metals in pig feed, fresh manure and wastewater and sediment from pond inflow and outflow of farms with one pond, two ponds, two ponds with separator and three ponds in series were investigated. Results indicate that heavy metals content in manure were in decreasing order of Cu>Zn>>>Cr>Pb>Ni>Cd and they were highly correlated with the feed (r=0.90). Reduction of heavy metals in the wastewater was in decreasing order of Cu(62%)>Zn(36%)>Ni(34%)>Pb((31%)>Cd(16%)>Cr(9%) and the reductions were significantly higher in the 3-pond system than the other systems. Copper and zinc were the highest in concentrations in the pond sediment (833 and 655 mg/kg respectively) whereas the other heavy metals ranged from 17 to 81 mg/kg. Reductions of heavy metals in the sediment between inlet and outlet sediment of the 3-pond system (47%) was significantly higher than the other three systems (<16%). The concentration of Cd in the outflow of all the pond system studied exceeded the 0.02 mg/L Malaysian standard set for the discharge downstream of water intake indicating its mobility and thus the need of other methods to polish the effluent for compliance.

Key words: Animal feed · Heavy metals · Oxidation Pond · Wastewater treatment · Retention

INTRODUCTION

Heavy metals have drawn the attention of the public in recent years due to their toxicity when present in excess and their long-term persistence [1]. According to the estimate of Pirkle [2] lead poisoning affected more than 800,000 children between the age of one and five in the USA. Root, leaf and fruit crops irrigated with sewage were found to be contaminated with heavy metals such as Pb, Cd, Se and As [3]. Azeez *et al.* [4] reported high accumulation of heavy metals in the soil due to nine years of animal waste deposition. A study of feed and manure heavy metals conducted in England and Wales indicated that a range of heavy metals were present in animal feeds and manure [5]. Thus, it is important to study the removal of those heavy metals in animal waste treatment systems.

Oxidation ponds are common domestic and animal farm waste treatment systems in developing countries due to its low cost and maintenance and simple operation [6, 7]. Though they are mainly designed for the removal of solids, they also contribute to the removal of nutrients and bacteria. Many studies have been conducted on the effectiveness of such system in reducing organic solids, nutrients and bacteria [6, 8, 9]. Studies on their effectiveness in treating heavy metals have been reported and the results were mixed. Furthermore, most of the studies were conducted on industrial, domestic wastewater or sewage [10- 15]. Juanico *et al.* [14] reported the removal of 20-75% of heavy metals (Cu, Zn, Cr, Pb, Al) in two reservoirs in series used for seasonal storage of wastewater effluents for irrigation. High removal of heavy metals (Cu, Zn and Pb) in waste stabilization ponds and high rate ponds for domestic waste treatment system in Morroco was reported by Toumi *et al.* [13]. Studies of sediment heavy metals at retention ponds inlet and outlet draining retail and residential areas indicated a mixture of decrease and increase in concentrations [12]. Achoka [11] reported insignificant decrease in Cr, Cu and Ni in pulp and paper mill effluent oxidation pond treatment system. A study on the sewage treatment oxidation pond in Nigeria showed that it is capable of reducing some of the metals studied to some extent but not at desirable levels for discharge [10].

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In the tropical country of Malaysia, plenty of water is used to wash and cool the animal and flush the waste [16] and farm operators are required to treat the wastewater in oxidation ponds. Such raw wastewater is high in solids, organic matter and nutrients such as nitrogen and phosphorus [17]. Different oxidation pond systems and management have been reported to give different efficacies in the reduction of organic matter and nutrients [9]. However, the fate of heavy metals in animal waste oxidation ponds with different number of ponds has not been investigated. Ling *et al.* [18] reported that the tributary that received pig farm effluent has the highest mean copper concentration. According to Weis and Weis [19], wetland sediments are a sink for metals. Therefore, heavy metals in pig farm wastewater could potentially be retained in the sediment of the oxidation ponds. Therefore, the objectives of this study were to investigate the status of heavy metals in feed and manure and the performance of different pond systems in heavy metals retention.

MATERIALS AND METHODS

Samples Collection: Samples of pig feed, manure, pond sediment and wastewater were collected from four pig farms between September and December 2004 in Kota Samarahan Division, Malaysia. The farms have different oxidation pond systems treating wastewater, namely, one-pond system (1P), two-pond system (2P), two-pond with solid-liquid separator system (2PS) and three-pond system (3P). The standing pig population (SPP), the dimension of the ponds and the sizing of the first pond in relation to animal weight of the farms are given in Table 1. Composite samples of wastewater and sediment were collected at the inflow of the first pond and outflow of the last pond. Temperature and pH of the wastewater were measured *in situ* using a pH meter (Cyberscan pH100). Wastewater samples were collected using 2-L polyethylene bottles that have been previously soaked in 10% HNO_3 for 72 h and rinsed with deionised water. Sediment collected was stored in polyethylene bag and both wastewater and solid samples were stored in an icebox before being transported to the laboratory for analysis.

Samples Preparation and Analysis: Wastewater was filtered and preserved by adding HNO_3 to pH < 2 before analysis according to Standard Methods [20]. For the solid samples, the feed, manure and sediment, the samples were air dried for 2 weeks before grinding using mortar and pestle. All ground solid samples were sieved using a 63-micrometer sieve (USA Standard Testing Sieve). Dry matter content of the solids samples was determined by oven drying at 105 °C.

Twenty milliliters of a mixture of nitric acid (69%) and hydrochloric acid (37%) (1:3 HNO_3/HCl) was added to an air-dry sample (0.5 g for sediment and 3 g for feed) in a conical flask [20]. The mixture was digested on a hot plate until the solution was clear and volume reduced to less than 5 ml. It was cooled and rinsed with deionised water before filtration using a 45 μm glass microfibre filter. The filtrate was transferred to a volumetric flask and topped up to 50 mL before storing in polyethylene bottles to be analyzed by a Flame Atomic Absorption Spectrometer (Perkin Elmer 3110). Calibration solutions were freshly prepared from individual element stock standard solutions (1000 mg/l). Heavy metals determined were copper (Cu), zinc (Zn), cadmium (Cd), chromium (Cr), nickel (Ni) and lead (Pb). All samples were determined in triplicates.

Statistical Analysis: Significant difference in mean feed and manure heavy metals concentrations and reduction in concentrations in water and sediment among the farms and among type of heavy metals was analyzed using Univariate ANOVA. Tukey's method was used for multiple comparisons. All analyses were performed using SPSS 14.0.

Table 1: Standing pig population (SPP), order of ponds in series, pond dimensions of the three waste treatment systems and sizing design of the first of oxidation pond

System	SPP	Order of ponds	Pond DimensionL(m)xW(m)xD(m)	Design(m^3/45 kg animal)
1P	1330	1	15.6x9.4x9.4	0.80
2P	920	1, 2	20x17x2.7,	
			20x10x2.7	0.92
2PS	1200	1, 2	13.3x13.3x2.7,	
			13.3x13.3x2.7	0.31
3P	1920	1, 2, 3	20x13.3x3.3,	
			20x13.3x3.3,	
			13.3x13.3x3	0.35

RESULTS AND DISCUSSION

Feed and Manure: Mean concentrations of heavy metals in the feeds from the farms with different pond systems are given in Table 2. The mean concentration of Cu was the highest ranging from 90 to 328 mg/kg followed by Zn (117-122 mg/kg). The other heavy metals were much lower in concentrations (1-14 mg/kg) in the decreasing order of Cr>Pb>Ni>Cd and they were not significantly different from each other (P=1.0). Comparisons among farms showed that there was no significant difference in feeds heavy metals (P=0.189) among the farms. The source of Cu and Zn in the feeds was likely the added copper sulphate and zinc oxide where Cu served to suppress bacterial action in the gut and maximize feed utilization and Zn for curing scour [5]. The feed Cu concentration from all farms except the 2PS were higher than the maximum concentration reported by Nicholson *et al.* [5] (85-217 mg/kg) but the Zn concentrations were lower than that reported (173-986 mg/kg respectively). For other heavy metals, the levels were mostly higher than those reported (<5 mg/kg) in England and Wales [5]. The difference could be attributed to the different amount of minerals additives incorporated in the feed. Mineral additives have been reported to be relatively high in Pb and Cr other than Zn and Cu [5]. Besides that, plant constituents of the feed may contribute to heavy metals content due to their uptake from agricultural materials such as fertilizers, pesticides, composts and manures, sewage sludge and corrosion of metal objects [21].

Cu was also the highest in concentration in the manure followed by Zn and they were much higher than Cr, Ni, Cd and Pb (Table 3). Compared with the feed, concentrations of heavy metals was much higher in the manure, where the mean concentration of Cu, Zn and Pb in the manure was about 4 times of that in the feed, Cr and Ni 6-7 times and Cd, 8 times. Manure heavy metals is strongly correlated with the amount in the feed with Pearson's correlation coefficient of 0.90 (P<0.0005, N=48). Nicholson *et al.* [5] also reported correlations of heavy metals between manure and feed. Concentrations of Cu in manure in all the farms except the 2PS farm was higher than that reported by Nicholson *et al.* [5] (<1-807 mg/kg) in England and Wales but for Zn it is within the values reported (<5-2500 mg/kg).

Waste water: In the present study, temperature of the wastewater ranged from 27 to 30°C and pH ranged from 6.6 to 8.4. In the inflow wastewater, concentrations of heavy metals were low, less than 1 mg/L (Fig. 1a). Concentrations of Cr was the highest, between 0.5 mg/L and 1.0 mg/L, followed by Zn, Cd, Pb, Ni and Cu in that order which were all less than 0.2 mg/L. In the outflow wastewater, comparing among heavy metals, similar trend was observed where Cr was the most concentrated and Cu was the least (Fig. 1b).

The low concentrations of Cu in the wastewater were most likely due to sedimentation of Cu together with solids. Cu is more strongly complexed by abundant organic matter compared to the other heavy metals and

Table 2: Mean concentrations of heavy metals in feeds from the farms with different pond systems

System	Concentrations (mg/kg dm)						
	Cu	Zn	Cd	Cr	Ni	Pb	Mean
1P	327.8±12.1	121.4±1.1	1.8±0.2	13.5±2.0	6.0±1.0	5.1±0.9	38.6±53.3[a]
2P	280.9±8.3	117.2±1.7	4.5±3.1	6.9±4.3	7.9±2.5	5.6±0.5	79.3±30.2[a]
2PS	90.0±8.5	122.1±2.3	1.1±0.1	5.7±0.9	4.5±0.5	8.3±1.5	65.0±101.3[a]
3P	250.5±13.7	117.4±3.7	5.2±1.7	6.8±0.8	5.2±0.3	4.8±0.7	70.5±112.2[a]
Mean	237.3±103.2[a]	119.5±2.6[b]	3.1±2.0[c]	8.2±3.5[c]	5.9±1.5[c]	6.0±1.6[c]	

*Means in the same row/column with the same letters are not significantly different at 5% level of significance

Table 3: Mean concentrations of heavy metals in manure from the farms with different pond systems

System	Concentrations (mg/kg dm)						
	Cu	Zn	Cd	Cr	Ni	Pb	Mean
1P	1185.5±18.5	582.2±5.4	12.5±1.3	50.0±3.3	35.7±1.8	21.2±0.8	314.5±480.7[a]
2P	811.3±3.4	368.7±2.1	36.8±3.1	51.8±2.6	36.0±1.9	23.5±1.6	221.4±318.1[ab]
2PS	413.8±46.2	423.0±53.6	11.7±1.3	42.7±6.4	32.3±7.6	17.5±3.3	161.6±211.5[b]
3P	935.0±10.6	519.0±4.6	35.7±0.6	71.8±8.6	39.2±13.0	23.7±2.8	270.6±378.6[ab]
Mean	843.5±19.7[a]	474.1±16.4[b]	24.2±1.6[c]	53.1±5.2[c]	35.8±6.1[c]	21.5±2.1[c]	

*Means in the same row/column with the same letters are not significantly different at 5% level of significance

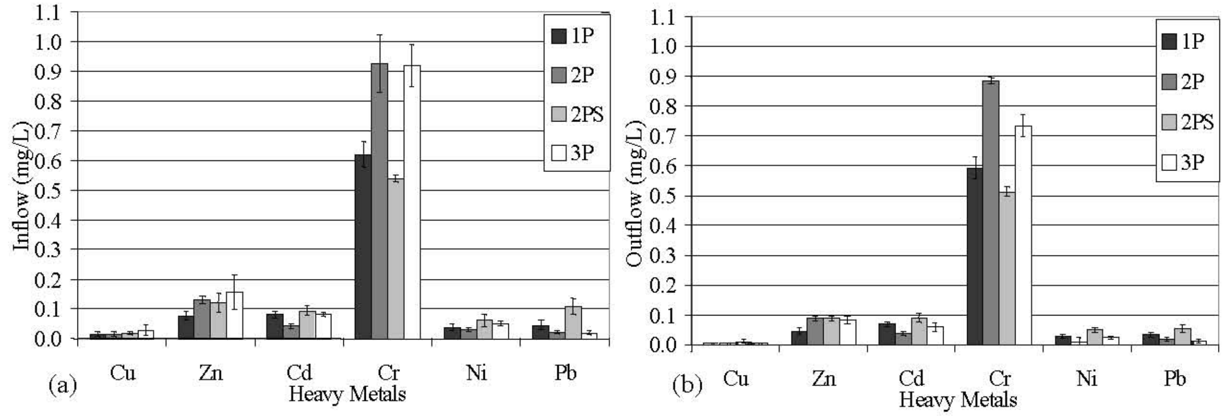

Fig. 1: Concentrations of heavy metals in inflow (a) and outflow (b) of the oxidation ponds. Error bars represent the standard deviation of replicate (*n* = 3) wastewater samples

Table 4: Reduction in the concentrations of heavy metals from inflow to outflow wastewater in different pond systems

	Reduction (%)						
System	Cu	Zn	Cd	Cr	Ni	Pb	Mean
1P	60.0±14.1	40.4±1.8	16.2±0.6	4.2±1.1	23.9±5.5	22.3±1.2	27.8±18.5[a]
2P	66.3±5.3	31.7±4.1	16.3±5.3	4.5±2.3	42.3±0.8	18.1±9.8	29.8±19.3[a]
2PS	41.7±11.8	26.3±6.8	3.6±0.6	5.0±3.7	18.0±6.6	47.4±12.7	23.7±21.1[a]
3P	78.8±12.4	46.3±10.9	26.8±2.5	20.1±6.0	53.3±4.6	34.3±8.1	43.3±21.5[b]
Mean	61.7±16.7[a]	36.2±9.7[b]	15.7±9.1[cd]	8.5±7.7[d]	34.4±15.5[b]	30.5±14.0[bc]	

*Means in the same row/column with the same letters are not significantly different at 5% level of significance

removal could occur by sorption at soil mineral surfaces and occlusion or co-precipitation by silicate and non-silicated clays [22]. Overall, mean heavy metals concentrations in the outflow were significantly lower than inflow (P=0.049) and Tukey's multiple comparisons did not detect any significant difference in heavy metals among the farms (P=0.110). Among the heavy metals, the mean concentration of Cr was significantly higher than the other heavy metals (P<0.0005) but there was no significant difference among Zn, Cd, Ni and Pb (P=0.129). Cu was significantly lower than Zn (P=0.014) but not significantly different from Cd, Ni and Pb (P=0.284). The high Cr in wastewater could be attributed to the Cr(VI) form instead of Cr(III). This is because pH recorded in the present study ranged from 6 to 8 and according to McGrath [23], complete precipitation of Cr (III) occurs below pH 5.5 and above that Cr(VI) is the more stable form in equilibrium with oxygen.

The reduction of heavy metals in wastewater indicated that the 3P farm showed significantly higher reduction compared to other systems (0.018<P<0.0005) and there was no significant difference among the other three systems (P=0.484) (Table 4). Among the heavy metals reduction, Cu was reduced the most (62%), followed by Zn (36%), Ni (34%), Pb (31%), Cd (16%) and Cr (9%) in that order. However, reduction of Zn, Ni and Pb were not significantly different (P=0.890) due to large variations. Reduction of Cd and Pb were not significantly different (P=0.078) and Cd and Cr were also not significantly different (P=0.743). Chipasa [24] reported similar trend of removal of heavy metals from industrial and municipal wastewater treatment system whereby Cu and Zn were removed more than Cd and Pb. Toumi *et al.* [13] reported higher removal rate of Cu, Zn and Pb in three domestic wastewater stabilization ponds in series, 92, 91 and 71% respectively. This difference is due to the setup as their system of domestic wastewater ponds were preceded with pre-treatment with screens, sand trap and oil/grease separator before the ponds. Thus, the ponds were not loaded by solids as in the present study where efficiency of the ponds may have been reduced by solids. Precipitation as insoluble compounds and adsorption especially with organic matter most likely led to the reduction of metals [25]. Pb for example also occurs as insoluble precipitates (phosphates, carbonates and hydroxyl-oxides) in the ponds [26].

Table 5: Concentrations of heavy metals in pond sediment at inlet and outlet in different pond systems

		Concentrations (mg/kg dm)						
System	Location	Cu	Zn	Cd	Cr	Ni	Pb	Mean
1P	Inlet	1492.5±19.4	807.0±1.3	11.8±0.3	54.8±8.0	41.3±0.3	89.2±1.5	416.1±608.7[a]
2P	Inlet	328.3±3.8	719.5±12.4	15.0±0.6	57.0±1.0	35.2±1.3	87.0±2.3	206.1±275.5[b]
2PS	Inlet	826.8±5.2	639.3±18.1	10.0±1.3	172.7±19.3	38.0±0.9	79.7±0.8	294.4±349.3[ab]
3P	Inlet	947.7±31.3	749.2±15.1	60.5±3.1	39.8±0.3	29.8±2.3	70.8±2.6	316.3±417.2[ab]
Mean	Inlet	897.5±480.1	728.8±69.8	24.3±24.2	81.1±61.5	36.1±4.9	81.7±8.3	
1P	Outlet	1310.3±18.9	749.8±3.4	10.5±0.6	51.7±3.7	41.7±0.8	87.7±1.3	375.3±537.9[a]
2P	Outlet	264.7±15.0	635.5±7.2	11.8±0.8	37.5±0.9	33.8±3.5	85.3±1.3	178.1±242.3[ab]
2PS	Outlet	1343.8±31.7	642.5±0.9	7.5±1.0	144.3±12.7	40.5±1.6	83.8±0.6	377.1±528.2[a]
3P	Outlet	155.7±2.8	295.0±4.4	10.7±0.8	30.7±0.6	24.8±1.1	63.8±1.3	96.8±31.6[b]
Mean	Outlet	768.6±646.5	580.7±197.5	10.1±1.8	66.0±52.9	35.2±7.7	80.2±11.0	
Mean	Inlet & Outlet	833.1±509.3[a]	654.7±163.0[a]	17.2±16.9[b]	73.6±60.5[b]	35.6±8.6[b]	80.9±14.6[b]	

*Means in the same row/column with the same letters are not significantly different at 5% level of significance

Table 6: Reduction in the concentrations of heavy metals in pond sediment from inflow to outflow in different pond systems

	Reduction (%)						
System	Cu	Zn	Cd	Cr	Ni	Pb	Mean
1P	12.2±6.2	7.1±5.3	11.3±2.4	5.8±0.5	-0.8±9.1	1.7±1.2	6.3±6.1[a]
2P	18.1±5.3	11.7±4.1	21.1±5.3	34.2±2.3	3.8±0.8	1.9±9.8	15.9±14.2[a]
2PS	-62.5±3.8	-0.5±6.7	25.0±6.1	16.4±14.4	-6.6±2.3	-5.2±0.9	-5.7±30.1[a]
3P	83.6±3.2	60.6±0.1	82.4±1.9	23.0±15.0	16.8±3.4	9.9±1.7	47.0±31.6[b]
Mean	12.9±59.8[ab]	19.7±27.7[ab]	34.9±32.1[a]	19.9±11.9[ab]	3.3±9.[9ab]	2.1±6.9[b]	

*Means in the same row/column with the same letters are not significantly different at 5% level of significance

Other than Cd (>0.02 mg/L), all the heavy metals in the outflow wastewater complied with Standard B of the Malaysian Environmental Quality (Sewage and Industrial Effluents) Regulations of 1979.

Sediment: At the inlet and outlet pond sediment Cu and Zn were the highest and Cd the lowest in concentrations (Table 5). The high concentration of Cu and Zn in the sediment corresponds with the high concentration in the feed and manure. In the inlet sediment, the 1P farm recorded the highest Cu followed by 3P, 2PS and 2P farm. This reflects the SPP and oxidation pond design whereby farm 2P has the lowest SPP and highest sizing design (Table 1). For the outlet, 2PS and 1P systems recorded very high Cu and 3P system recorded the lowest Cu. Even though 3P farm has higher SPP than 1P farm, the concentrations of Cu in both inlet and outlet sediments were lower than that of 1P farm. Similar observation was found for Zn where the 3P system showed the lowest outlet concentrations. This is possibly due to the presence of only one pond in 1P farm compared with the three ponds of 3P system which has two additional ponds that enable further retention of Cu and Zn.For the 2PS system, the inlet Cu, Zn and Cr were lower than outlet concentrations. This is likely due to the separation of solids from the liquid at the inlet. However, the outlet concentrations were still high due to the ponds being filled with sludge before installation of the separator. Overall, 2P system has the lowest and 1P system the highest mean heavy metals concentrations. Statistical analysis indicated that the mean concentrations of Cu and Zn were significantly higher ($P<0.0005$) than Cd, Cr, Ni and Pb concentrations which were not significantly different ($P=0.954$). Among the systems, 2PS showed no significant difference in mean heavy metals compared to other systems ($P=0.087$) and the 1P system was significantly higher than 2P and 3P systems ($P=0.006$ and 0.012 respectively). This could be due to the much lower sizing design of 2PS system and even though there was a separator, the ponds were already filled with sludge prior to its installation.

Comparing between outlet sediment concentrations with the inlet, reduction in concentrations was generally low for the one and two ponds systems with 1P system recording the lowest reduction (Table 6). For Ni, the outlet concentration was even higher than inlet giving negative reduction. This is most likely due to the anaerobic digestion of sludge where organic matter were decomposed by bacteria to produce gases such as methane, carbon dioxide, nitrogen and hydrogen sulfide which were released to the atmosphere but the determination of heavy metals was conducted in dry weight basis [24]. For the 2PS system, the negative reduction of Cu, Zn, Ni and Pb indicates the positive effects of solid liquid separator whereby heavy metals that are highly sorbed to solid organic matter are retained in the solids resulting in low concentrations at pond inlet. The 3P system showed the highest percent reduction and the mean reduction was significantly different from the other systems (P=0.049). For the 3P system, with the addition of one more pond, besides further precipitation and sorption and the bacteria in the anaerobic digestion of the first and second ponds, facultative bacteria and algae present in the facultative third pond further took up heavy metals resulting in the highest reduction. Biosorption, bioaccumulation and sorption to extracellular biopolymers were possible mechanisms of microbial uptake in the pond [24].

CONCLUSIONS

The manure heavy metals concentrations were highly correlated with that in the feed. There was significant reduction of heavy metals in wastewater in all the pond systems studied. In the inflow and outflow wastewater, concentrations of Cr were the highest indicating its mobility. High concentrations of Cu and Zn in the sediment and low concentrations in the water indicate the efficient retention of those heavy metals in pond sediment. The 3P system performed significantly better than all the other systems in reduction of heavy metals in wastewater and sediment. The presence of solid-liquid separator was found to reduce inlet sediment heavy metals concentration. Effluent heavy metals concentrations of all systems except cadmium complied with the standard for discharge downstream of water intake stipulated by the Malaysian Environmental Quality (Sewage and Industrial Effluents) Regulations, 1979. Other treatment methods are required to ensure outflow cadmium compliance (=0.02 mg/L).

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