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Characterization of Malaysian sewage sludges and nitrogen mineralization in three soils treated with sewage sludge

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ABSTRACT

Studies to determine the chemical composition of sewage sludges produced in Malaysia and potentially mineralisable nitrogen (N_o) and mineralization rate constant (k) of sewage sludge in three Malaysian soils are reported. The sludges collected from ten wastewater treatment plants in Malaysia were acidic in nature and the N, P, K, Ca and Mg contents were variable. The heavy metal (Pb, Cd, Cu, Mn and Ni) concentrations of the sludges, except for Zn, were below the European Union Maximum permitted level in sludges. In an incubation study, three topsoils of Bungor, Jawa and Serdang series were treated with three rates (0, 140 and 420 kg N ha-1) of dewatered sewage sludge and incubated about 60% of the water holding capacity for 12 weeks. Mineralization of N exhibited a slow initial rate, followed by a rapid increase in rate in week 4 to 8. Accumulation of mineral N ranged from 50.5 to 147.6 mg kg^{-1} soil. Bungor and Jawa series had higher N mineralization than Serdang series. Sludge added at 420 kg N ha^{-1} resulted in the highest concentration of net mineralised N. Values of potentially mineralisable N, (N_o), and mineralization rate constant, (k), ranged from 23.4 to 137.5 mg N kg^{-1} soil and 0.036 to 0.082 week-l, respectively. It was concluded that N mineralization of the sewage sludge treated soils was dependent on the application rate of sludge and soil type.

Keywords : Sewage sludge characterization, nitrogen mineralization.

INTRODUCTION

Indah Water Konsortium Sdn. Bhd.(IWK) (1997) had reported that Malaysia produce about 5 million m^3 of sewage sludge per year. On 9th December 1993, the Government of Malaysia handed over the national sewerage privatization project to IWK to manage a more modern and efficient sewerage system for the country. Currently, IWK operates and maintains over 4,300 public sewage treatment plants all over Malaysia, desludges and treats sludges from over 0.8 million septic tanks regularly and monitors effluent samples from sewage treatment plants to ensure they meet the Department of Environment's standards. In recent years, industrial wastes and municipal wastewater sewage sludge or biosolids all over the world have been applied regularly to soil (Krogman *et al*. 1997; Benton and Wester, 1998). They have large nutrients values, mainly in N, P and K and their application to land is a way of using the sludges as a fertilizer (Coker *et al*., 1987 and Kulling, 2001). According to Pagliai *et al*. (1981) and Smith *et al*. (1992),

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organic matter in sewage sludge can also increase crop productivity by improving soil physical and chemical properties. Fifty to 90 % of the N in sludge is in the organic form (Sommers, 1980). Information on the rate of mineralization rates of organic N in sludge is necessary to predict N availability during a cropping season. However, mineralization of organic N in sewage sludge is a complex process, and is dependent on several factors such as soil type, rate and type of sludge applied, soil pH, temperature, aeration and moisture (Terry *et al*., 1981 and Mtambanengwe *et al*., 2004). Most studies of N mineralization of sewage sludge have been carried out mainly in temperate soils. Information on N mineralization of sewage sludge in the highly weathered acidic soils of Malaysia is limited.

In year 2000, a project was undertaken by the Department of Land Management, Universiti Putra Malaysia, to study the potential use of sewage sludge as a fertilizer for corn cultivation. This study was a part of the long-term project to determine the chemical composition of sewage sludges produced by IWK and N- mineralization potential of a selected sludge in the three soil types.

MATERIALS AND METHODS

Sludge Characterization

Sewage sludges were collected from ten different treatment plants throughout Peninsular Malaysia with the co-operation of IWK to represent the various types of sludges produced. The sludge samples were air-dried and then ground to pass a 1 mm sieve for chemical analyses: pH, organic C, total N, heavy metals and macronutrients content. Organic C was determined according to the combustion method (McKeague, 1976). One gram of sewage sludge was placed in a crucible and put into a furnace at 350 °C for an hour. The temperature was then raised to 550 °C and left for 24 hours. The remaining ash was weighed and organic C was calculated from the loss in weight during ashing. Total N was determined using the Kjeldhal method (Bremner and Mulvaney, 1982). Total analysis of the heavy metals and macronutrients were determined using the aqua-regia method. The extraction solution was made using HCl and HNO_3 solution (3:1). Heavy metals (Pb, Cd, Cu, Ni, Mn, Zn, and Fe) and macronutrients (Mg, Ca and K) in the solution were determined using PE 5100 atomic absorption spectrophotometer.

TABLE 1

Physical and chemical properties of the three Malaysian topsoils used in the incubation study.

Soil Series	Size fractions % clay	silt	sand	Soil Texture Class	pH H_2O	pH (KCl)	% Org. C	% Total N	C/N
Bungor	28.4	6.9	64.5	Sandy clay loam	4.9	3.6	2.2	0.188	11.9
Serdang	35.8	14.2	50.0	Sandy clay loam	3.6	5.7	1.3	0.154	8.60
Jawa	21.5	71.4	7.0	Silt loam	4.0	3.2	6.4	0.376	17.4

Mineralization of Sewage Sludge

A laboratory experiment was conducted to determine N-mineralization potential of a sewage sludge applied to different soil series in an incubation period of 12 weeks. Topsoils of Bungor (Ultisols), Jawa (Inceptisols) and Serdang (Ultisols) series were applied with dewatered sewage sludge (8 month old) at 0 %, 100%, 300 % of recommended fertilizer N rate for maize cultivation (0, 140 and 420 kg N ha^{-1}). Physical and chemical characteristics of the three soils and sewage sludge are as given in Table 1. Treatments were laid out in complete randomized design and replicated 4 times. The sludge used in the incubation study had a pH of 5.0, and contained 28.4% organic carbon, 37.9 mg kg^{-1} mineral N, 2.0 % total N, 0.21% P and a C/N ratio of 13.6.

Three hundred grams of air-dried soil was placed in a plastic container and mixed with 3 rates of sewage sludge i.e. 0, 0.5 and 1.5 g (0, 140 and 420 kg N ha^{-1}) of sludge and adjusted to about 60% water holding capacity by regular weighing and addition of distilled water to compensate for the evaporation loss. The container was closed with a parafilm to enable humified free air to continuously pass over the soil-sludge mixture. The soils were incubated in a dark cupboard at room temperature of 24o C. At week 0, 1, 2, 4, 6, 8 and 12, a sub-sample of 10 g was removed for extraction of mineral N with 40 ml of 2 M KCl. The sample was shakened for an hour, then 10 ml of the filtered extract was distilled with MgO for NH^{+}_{4}-N and Devarda's alloy for NO^{-}_{3}-N and NO^{-}_{2}-N, and collected in boric acid. Titration was done with 0.0025 M HC1.

The mineral N data were then statistically analyzed using a non-linear regression approach described by Smith *et al*. (1980). Estimates for the amount of potentially mineralizable N (N_o) and the first-order rate constant (k) for the 12 weeks incubation period were obtained using the following equation:

$$N_m = N_o (1 - e^{-kt})$$

Where:

N_m = amount of N mineralized at a specific time

N_o = potentially mineralisable N

k = first – order rate constant

t = time of incubation

RESULTS AND DISCUSSION

In general, the ten collected sludges were acidic ranging from pH 3.57 to 6.43, because no lime was used in the treatment of the wastewater at the treatment plants (Table 2). Higher pH values of sludges in other countries had been reported (Serna and Pomeras, 1992; Nielson *et al*., 1998; Moreira et al 2008) due to the addition of CaO and $CaCO_3$ during the sludge treatment. Organic carbon content varied greatly from 6.13% to 56.67% and the highest was in sludge 5 (mixture of light industry & domestic type). The variation in organic C seemed to be independent of sludge type. The organic C content in these sludges were higher than those reported by Hsieh *et al*., (1981), Parker and Sommers (1983), Chae and Tabatabai

TABLE 2
Nutrient contents and pH (n=3) of sewage sludges taken from ten different wastewater treatment plants.

Sewage sludge	pH (H_2O)	%C	%N	C/N	%P	%Ca	K	Mg
							---- mg kg^{-1} ------	
1	4.45	48.21	1.67	28.87	0.238	0.627	1205	445
2	3.92	6.13	0.68	9.03	0.321	0.422	1021	521
3	6.43	32.10	1.54	20.84	0.410	1.17	941	1690
4	3.92	11.15	2.70	4.13	0.652	0.323	462	278
5	3.57	56.67	1.52	37.94	0.821	0.160	401	389
6	5.61	41.36	2.27	18.22	0.778	1.12	1209	112
7	5.24	37.41	2.82	13.26	0.634	0.832	728	870
8	5.89	18.28	2.65	6.90	1.20	2.16	621	2062
9	6.02	33.35	2.90	11.50	1.62	0.926	972	2902
10	5.72	41.23	2.64	15.62	0.470	0.640	678	1575
Mean	5.08	32.59	2.14	16.63	0.714	0.838	824	1084

Sewage sludges 1, 2, 3, 4, 6, 7 and 10 – domestic type
Sewage sludges 5, 8 and 9 – mixture of light industry & domestic type

(1986). Total N content ranged from 0.68 to 2.90 %, whereas P was from 0.24 to 1.62 %. Total N content of sludges in this study was higher than reported by Lindemann and Cardenas (1984), Indah Water Konsortium Sdn. Bhd. (1997) and Moreira *et al*. (2008). The C/N ratio of the sludges ranged from 4.1 to 38.0 with a mean of 16.6. The range of Mg and K concentrations were from 278 to 2902 mg kg^{-1} and 401 to 1209 mg kg^{-1}, respectively. The concentration of Ca varied from 0.16 to 2.16 %. These macronutrients are beneficial for plant growth and the amount in the sludges are of considerable value as a fertilizer replacement. The concentrations of micronutrients reported in this study were similar to those reported by Kala (1998), Phuah (1999) and Christina (1999). The concentrations of Pb, Cd and Cu varied from 36 to 308 mg kg^{-1}, 0.51 to 6.49 mg kg^{-1} and 63 to 732 mg kg^{-1}, respectively and the concentration of Mn and Ni ranged from 32 to 420 mg kg^{-1} and 10 to 151 mg kg^{-1}, respectively (Table 3). Sludge 9 (light industry and domestic type) had the highest concentrations of heavy metals (Pb, Cd, Cu, Mn and Ni). The Fe concentrations in these sludges ranged from 1.22 to 4.01 % whereas Zn varied from 153 to 7012 mg kg^{-1}. According to the Commission of the European Communities 1986 guideline, the concentration of heavy metals in all the sludge samples did not exceed the maximum permitted concentration of heavy metals, except for the Zn content of sludge 8. The high Zn concentration may be due to the corrosion of galvanized iron pipes in the old sewerage system (Kumper, 1985) and toiletries such as shampoo (Comber and Gunn, 1994).

Nitrogen mineralization pattern of three application rates of sewage sludge in the three soils are shown in *Figs. 1, 2 and 3*. Accumulation of NH_4^+-N in Bungor series were about 80 μg N/g soil up to the 8th weeks and then decreased after 8 weeks while NO_3^--N concentration increased more rapidly. This indicates a rapid nitrification of NH_4^+-N after week 8. Chandeler (1984) reported that the decrease in NH_4^+-N was not always accompanied by an increase in NO_3^--N. This suggests N immobilizaton or denitrification could have occurred in these soils. In the Serdang and Jawa series, the NH_4^+-N concentration increased up to 12th week while nitrification was slower than Bungor series, although Serdang has similar topsoil

TABLE 3
Heavy metal contents (n=3) of sewage sludges taken from ten different wastewater treatment plants (mg kg^{-1}).

Sewage sludge	Pb	Cd	Cu	Ni	Mn	Zn	% Fe
1	36	1.57	127	19	101	841	3.22
2	51	0.51	112	20	127	892	4.01
3	47	3.21	141	26	362	997	2.62
4	46	1.22	63	10	32	153	2.54
5	121	2.51	696	15	73	454	2.27
6	60	5.78	110	13	132	1201	2.63
7	68	3.52	178	21	257	1322	1.90
8	189	6.02	223	38	282	7012	1.22
9	308	6.49	732	151	420	3778	3.31
10	72	3.22	190	15	105	3218	3.20
Mean	100	3.41	257	32	189	1986	2.69

Sewage sludges 1, 2, 3, 4, 6, 7 and 10 – domestic type
Sewage sludges 5, 8 and 9 – mixture of light industry & domestic type

TABLE 4
Estimates of N mineralization (N_0) and rate constant (k) using non-linear regression equation (mean n = 4)

Sewage sludge treatment rates	MeasuredTotal N net mineralized in 12 weeks	Predicted net N mineralized in 12 weeks (N_o) δ	Rate constant (k) δ	$N_0\,k$	R^2
kg Nha^{-1}	-------------- µg g^{-1} soil	---------------	week $^{-1}$	------ µg g^{-1} soil week^{-1}	------
		Bungor Series			
0	107.5	92.3	0.042**	3.88	0.977**
140	140.4	137.5	0.036**	4.95	0.979**
420	145.8	91.4	0.060**	5.48	0.988**
		Jawa Series			
0	105.5	98.5	0.053**	4.23	0.982**
140	127.8	108.8	0.042**	4.45	0.984**
420	147.6	124.9	0.042**	5.25	0.983**
		Serdang Series			
0	50.5	23.4	0.090**	2.11	0.982**
140	57.6	31.7	0.071**	2.25	0.985**
420	65.3	32.2	0.082**	2.64	0.985**

δ - N_0 and k obtained from non-linear regression equation: $N_m = N_0\,(1 - e^{-kt})$
R^2 - Correlation coefficients between measured and predicted total net mineralized N and k values obtained were significant at P<0.01 (**)

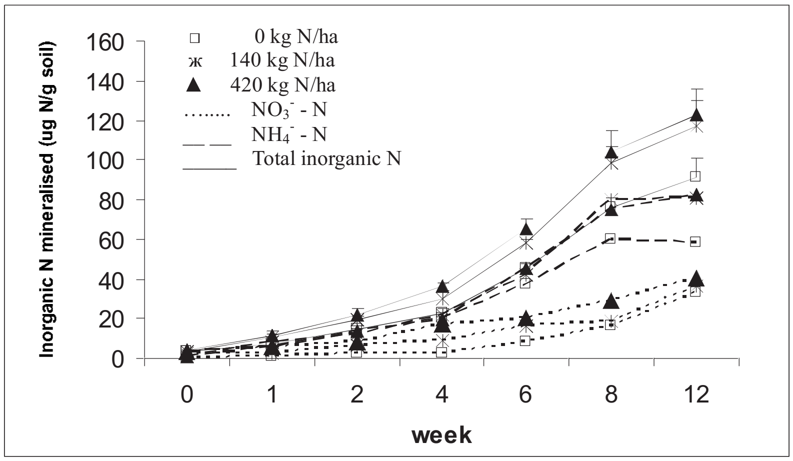

Fig. 1: Nitrogen mineralization of different application rates of sewage sludge in Bungor soil series (Bars indicate standard deviation of means).

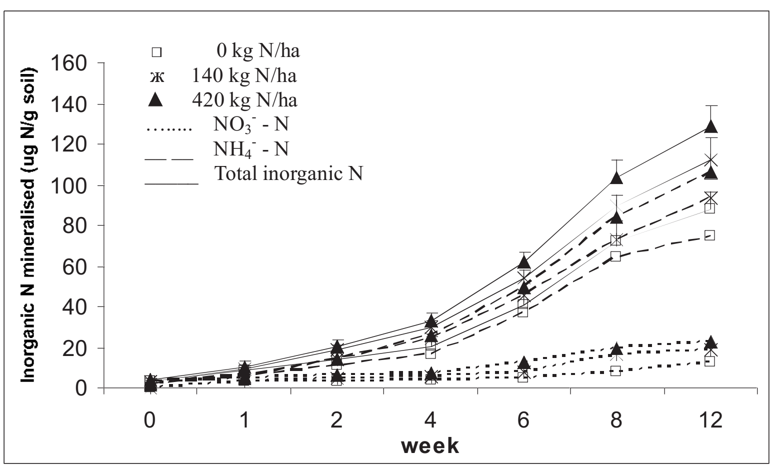

Fig. 2: Nitrogen mineralization of different application rates of sewage sludge in Jawasoil series (Bars indicate standard deviation of means).

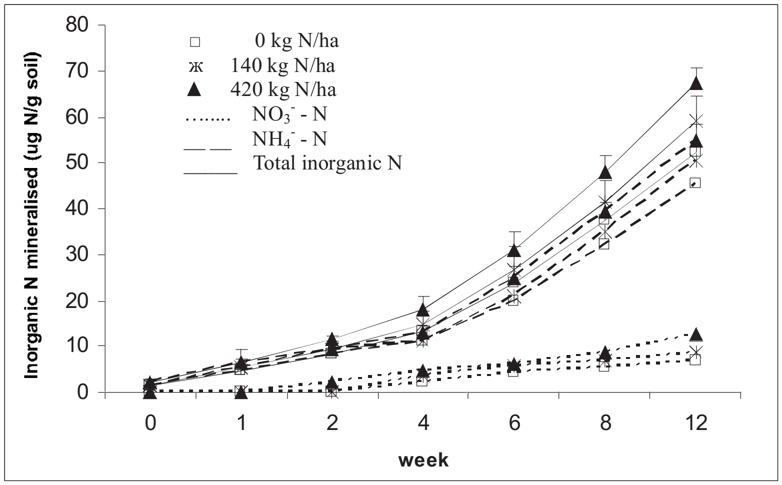

Fig. 3: Nitrogen mineralization of different application rates of sewage sludge in Serdang soil series (Bars indicate standard deviation of means).

soil texture as Bungor. In general the concentrations of mineral N were higher in both Bungor and Jawa soils and lower in Serdang series which had low organic C. The reason could be as stated by Mengel and Kirkby (1979), the bacteria responsible for the oxidation of ammonium to nitrate number only in thousands per gram of soil under poor conditions. Considering this, the population of nitrifying organisms in Serdang soil may have been very low at the beginning of the experiment.

The sludge rate of 420 kg N ha^{-1} gave the highest predicted or potentially mineralizable N (N_0) value in all three soils treated with sludge (Table 4). This is in agreement with the previous studies done by Magdoff and Chromec (1977) and Terry *et al*., (1981) that nitrogen mineralizaton in soil increases with an increase in sludge rate. The N_o and k (rate constant) obtained in Bungor, Jawa and Serdang series varied considerably, suggesting that *N* mineralization in soil treated with sewage sludge varied with the rate of sludge and the type of soil receiving the organic waste. Mean k values were lower than reported by most research. In this study, the k values did not increase with the sludge rate at most treatment levels. The values of N_o obtained for the Bungor and Jawa soils ranged from 91.4 to 137.5 mg kg^{-1} soil. For Serdang soil the values were smaller (23.4 to 32.2 mg kg^{-1} soil). The k (rate constant) values ranged from 0.036 to 0.090 week^{-1}. The N availability index (N_ok) of the sludges ranged from 2.11 to 5.48 mg kg^{-1}, with the lowest in the Serdang soil. The high mineralization rates occurred during week 4 to 8 of incubation, and are likely to be associated with the decomposition of labile organic N. After week 8 the more recalcitrant organic N appeared to predominate in the organic *N* pool, as mineralization rates slowed (Lindemann and Cardenas, 1984 and Alvarez and Alvarez, 2000). The low net mineralized N in Serdang soil compared to the Bungor and Jawa soils could be due to the low amount of organic matter and consequently a low pool of indigenous organic N. However, the organic matter in the Serdang soil had a low C:N ratio, suggesting a high potential mineralization potential. Nitrogen immobilization by the heterotrophic microorganisms of the soil may also be occurring (Smith *et al*., 1992). The correlation coefficients R^2 as shown in Table 4 were significant ($P<0.01$) and may be concluded that in all treatment there were good agreement between the observed points and calculated values of N mineralized throughout the incubation period.

CONCLUSIONS

Sewage sludges produced from Malaysian wastewater plants were acidic and have variable chemical compositions. The N-mineralization rate in sewage sludge treated soils is dependent on the sludge application rate and the chemical and physical characteristics of the soil receiving the organic waste. Higher mineralization rates with or without sludge addition were found in the Bungor and Jawa series. The Serdang series with lower pH and indigenous organic C had much lower mineralization potentials, even when large amounts of sludge were added. Thus, this study highlights the importance of understanding the properties and behavior of the soil in formulating any sewage sludge- based fertilizer programme for the supply of N for plant growth.

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