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The effect of ageing on the fertilizer value of sludge from Botswana

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ABSTRACT: This study was designed to characterize sludge of three different ages (36 months old, 3 months old and fresh sludge referred to as Type 1, Type 2 and Type 3 sludge respectively) in an endeavor to appreciate their suitability for use as manure for arable agriculture. Sludge properties including volatile solid and nutrient contents, concentrations of the heavy metals As, Cd, Cr, Co, Cu, Mn, Pb, Ni and Zn as well as coliform population were determined. The concentrations of plant nutrients in the sludge followed the order Type 3 sludge > Type 2 > Type 1 sludge with nutrient content scores of 1.4, 2.1 and 2.6 respectively. Scores for potential leachability of heavy metals from the three sludge were 2.1, 1.9 and 2.3 for Type 1, 2 and 3 sludge respectively, indicating that Type 2 sludge would release a higher concentration of heavy metals to the environment than the other two sludge types. The faecal coliform population in Type 3 sludge was higher than in Types 2 and 3 sludge with values of 5.6log$_{10}$MPN/10g dry solid, 4.6log10MPN/10g dry solid and 2.7log$_{10}$MPN/10g dry solid respectively. These results revealed that the nutrient contents and heavy metal concentrations in the three sludge types may not hinder their application to soil for arable purposes but steps to allow for further pathogen reduction need to be taken to reduce health risks. @JASEM

Application of sludge (a residue of organic and inorganic solids derived from wastewater treatment) to soil is emerging as an economically and environmentally acceptable alternative to disposal through landfill and incineration (Stacey *et al*., 2001) because of the agronomic benefits associated (Veeresh *et al*. 2003; Lindsay and Logan 1998). It is practiced in several countries in Europe (Tlustoš *et al*. 2000), America (Krauss and Page 1997), Asia (Wong *et al*. 2001), and Africa (El-Naim and El-Housseini 2002, Snyman *et al.* 2000). Sludge production in Botswana especially in Gaborone City has increased recently as a result of the rapid rate at which the city is growing and the introduction of advanced wastewater treatment techniques. There is therefore a need to put in place appropriate disposal methods that will maintain a healthy environment and guarantee plant, animal, and human health.

In Botswana, in an effort to increase arable farming around major settlements, funding is available to small horticultural projects and the trend is to use sludge. Considering the fact that soils in semi-arid regions like Botswana are generally poor in physical conditions like water retention capacity, and are characterized by low plant nutrient status, commercial fertilizers are required for any reasonable agricultural production. Whereas these fertilizers mainly serve to improve the plant nutrient status of the soil, sludge in addition to this, improves soil physical, chemical and biological properties (Lindsay and Logan 1998; Veeresh *et al*., 2003; Pascual *et al.*, 1997). Using sludge as manure could therefore improve the condition and fertility of these arid soils, while simultaneously converting waste into a resource. Its use as manure for the production is however constrained by five of its constituents namely pathogen load, heavy metal concentrations, plant nutrients contents, organic matter content usually measured as volatile solids, and the concentrations of toxic organic compounds (US EPA, 1999).

Previous studies on sludge from the Gaborone City Council wastewater treatment plant (Ngole *et al.* 2006) concentrated on the survival of sludge borne-coliforms in different soils. No study has evaluated the fertilizer value of this sludge even though it is currently being used as manure by some farmers. This study was designed to evaluate the quality of the sludge produced at this plant in terms of its heavy metals concentrations, pathogen load, nutrient contents and other physico-chemical and biochemical properties that may affect its fertilizer value. The effect of age on the fertilizer value was also investigated.

MATERIALS AND METHODS

Sludge Sampling: Three types of sludge differentiated by their ages (36 months old, 3 months, fresh sludge from the drying beds henceforth referred to as Type 1, Type 2 and Type 3 sludge respectively) were used in this study. Samples of Type 1 and Type 2 sludge were collected from the respective sludge piles around the treatment plant. Three piles each of Type 1 and Type 2 sludge were divided into 8 sections and 2 samples collected from each section. Samples of each sludge type were manually homogenized to form a composite sample which was representative of the particular sludge type. Type 3 sludge samples were collected from sludge drying beds which were about to be emptied onto the sludge piles. Sludge sampling from the drying beds was guided by methods described in US EPA (1999)

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where each drying bed is divided into 4 and 2 samples collected from the centre of each quarter, and at the centre of the bed.

Sludge Analyses: Sludge properties including solid content (SC), pH, electrical conductivity (EC), total dissolved salts (TDS), cation exchange capacity (CEC), exchangeable bases K, Na, Ca, Mg, organic matter (OM), total Kjeldahl nitrogen (TKN), available nitrogen (NH_4–N, + NO_3–N), and Olsen P were determined according to methods described by van Reeuwijk (1993) and USDA (1996). Chloride, sulphate, carbonate and bicarbonate concentrations were determined in the saturation paste extracts of each sludge sample (USDA (1996) whereas the volatile solid content (VS) was determined by ashing at 550ºC in a muffle furnace (Standard Methods for the Examination of Water and Wastewater 1996).

The US EPA’s 1992 toxicity characteristic leaching procedure (TCLP) was used to determine the concentrations of the potentially leachable fraction of heavy metals As, Cd, Cr, Co, Cu, Mn, Pb, Ni and Zn in the three sludge types. Each sludge sample was also extracted with aqua regia (3HCl + HNO_3) solution as described by Tokalioğlu *et al*., (2003) to determine the pseudo-total concentrations of these heavy metals. A SpectraAA Varian 220 FS flame atomic absorption spectrometer (FAAS) with deuterium background correction and equipped with a graphite furnace component (GTA 10) was used to determine the concentration of heavy metals in the different extracts. Enumeration of total coliforms (TC) and faecal coliforms (FC) in the different sludge types was done using the multiple tube fermentation procedure as described by US EPA (1999). A bioMérieux Sa API 20 E identification system, Software Package and Analytical Profile index Library were used to identify the different enteric bacteria contained in each type of sludge.

To compare the fertilizer value of the three sludge types, their properties were given scores on a scale of 1 – 3 with those scored as 1 indicating the lowest fertilizer value and 3 the highest. With regards to nutrients, the sludge type with the highest value for the specific nutrient was given the highest score (3) and that with the lowest value the lowest score (1). Considering the fact that a high concentration of metals and coliforms in the sludge would degrade its fertilizer value, the sludge type with the highest concentration of each heavy metal as well as faecal coliform was scored 1 and that with the lowest concentration scored 3. These scores were averaged and used to compare the fertilizer value of the three sludge types.

RESULTS AND DISCUSSIONS

Sludge Properties: Whereas the solid content of the sludge increased with age (Type 1 > Type 2 > Type 3), the VS and OM contents decreased (Type 3 > Type 2 > Type 1) as indicated by the values in Table 1. The higher content of solid materials in Type 1 sludge could be attributed to its age because it had been exposed to the desiccating action of the sun for a longer period than either of Type 2 or Type 3 sludge. The longer period of exposure also account for the lower values of OM and VS content in this sludge. Reduction in OM content of sludge with age has also been reported by Leifeld *et al.*, (2001) and is attributed to the processes of humification and mineralization of the sludge-derived OM (Sollin *et al.,* 1996). These processes convert organic material in the sludge to inorganic forms resulting in a reduction of the amount of OM accumulated with age. Volatile solid content in sludge correlates directly with the amount of OM contained in the sludge (US EPA, 1984). The observed reduction in OM with age may therefore explain the reduction observed in the VS content of the sludge with age.

Type 1 sludge was more acidic (pH = 5.4) than both Types 2 and 3 sludge with pH values of 7.1 and 6.6 respectively. Microbial decomposition of OM results in the production of organic acids among other compounds (Veeresh *et al.* 2003). The lower pH value obtained for Type 1 sludge compared to Type 2 and Type 3 sludge could be attributed to the accumulation of organic acids produced during the decomposition of the OM contained in the sludge. A lower sludge pH implies an increase in the tendency for ionic compounds to go into solution and consequently, a higher EC. Values for EC and TDS followed the order Type 1 > Type 2 > Type 3 sludge, indicating an increase in EC and TDS with age of sludge. This pattern correlates negatively with the values for pH (r = -0.90) and OM content (r = -0.94) of the sludge. About 80 % (R^2 = 0.81) of the changes observed in the EC of the three sludge types could therefore be explained by changes in pH and OM content in the sludge. According to EL-Naim & EL-Houseini (2002), OM humification results in the formation of carboxyl and phenolic functional groups onto which cations could be adsorbed. Due to the availability of nutrients, microbial activities are more vigourous in fresh than in old sludge, resulting in a faster rate of OM mineralization. More of the carboxyl and phenolic functional groups are likely to be present in the young sludge providing more sites onto which cations could be held thus explaining the higher value for CEC in Type 3 sludge (Table 1).

Table 1: Physico-chemical properties and nutrient content of the three sludge types

Properties	Type 1 sludge ± SD	Type 2 sludge ± SD	Type 3 sludge± SD
Solid content (%)	50.3 ± 9.2	47.7 ± 5.8	38.3 ± 4.5
VS (%)	23.0 ± 4.3	25.7 ± 2.6	29.2 ± 10.2
pH (H_2O)	5.4 ± 0.4	7.1 ± 1.1	6.6 ± 0.5
EC (mS/cm)	4.4 ± 0.4	3.4 ± 1.6	3.2 ± 1.6
TDS (mg/l)	3014.0 ± 233.6	2176.0 ± 104.7	2048 ±102.3
Cl^- (mg/kg)	52.4 ± 45.9	51.6 ± 16.4	40.2 ± 15.7
SO_4^{2-} (mg/kg)	2744.1 ± 121.8	2716.3 ± 167.9	1533.5 ± 92.2
HCO_3^- (meq/l)	9.9 ± 2.7	29.4 ± 4.4	31.8 ± 4.7
CEC ($cmol_c$/kg)	89.0 ± 3.2	93.5 ± 2.9	128.1 ± 7.8
Exch. Ca ($cmol_c$/kg)	35.8 ± 2.5	46.9 ± 0.8	41.0 ± 3.0
Exch. K ($cmol_c$/kg)	11.9 ± 1.7	16.5 ± 1.2	15.6 ± 2.0
Exch. Mg ($cmol_c$/kg)	37.9 ± 3.2	34.6 ± 4.1	64.2 ± 5.3
Exch. Na ($cmol_c$/kg)	19.1 ± 2.0	14.2 ± 3.1	13.0 ± 1.0
OM (%)	23.1 ± 12.8	32.4 ± 16.1	41.0 ± 14.6
Olsen P (mg/Kg)	7321 ± 920	7696.4 ± 571	9370.1 ± 780
TKN (%)	3.4 ± 2.1	4.5 ± 2.6	5.5 ± 2.0
NH_4-N (mg/Kg)	612.4 ± 236.4	614.4 ± 267.9	1369.4 ± 357.8
NO_3-N (mg/Kg)	546.2 ± 254.7	636.5 ± 227.2	1654.3 ± 253.8
Fe (%)	2.9 ± 0.4	2.0 ± 0.5	0.8 ± 0.4
Al (%)	3.1 ± 0.3	3.0 ± 0.1	1.7 ± 0.6

Sludge nutrient content: Due to the solubility of K and chloride compounds in water, more of these compounds are discharged with the treated effluent than are accumulated in the sludge derived from the wastewater, which may explain the low concentration of these ions in the sludge compared other organic manure. The concentration of exchangeable bases in the sludge followed the order Ca > Mg > Na > K in Type 1 sludge whereas in Type 2 and 3 sludge, the orders were Ca > Mg > K > Na and Mg > K > Ca > Na respectively (Table 1). Whereas the carbonates were not detectable, the concentrations of bicarbonate compounds decreased with increase in age of sludge (Table 1). The concentrations of Olsen P in the three sludge types followed the order Type 3 sludge > Type 2 sludge > Type 1 sludge, indicating a decrease with ageing of sludge. Whereas mineralization and leaching may account for the lower value of P obtained in Type 1 sludge, the higher content of OM in Type 3 sludge explains its higher P concentration since most of the P is not available for leaching. Phosphorus in young sludge exists mainly in the organic form, and becomes available for uptake or leaching only after it has been mineralized to inorganic forms (Maguire *et al.*, 2001).

Total Kjeldahl nitrogen concentration as well as the concentrations of NH_4-N and NO_3-N was highest in Type 3 than in the other sludge types (Table 1). Decrease of TKN and both NH_4-N and NO_3-N with age of sludge is attributed to volatilization and leaching of nitrogen containing compounds from the sludge (Gilmour and Skinner 1999). More N would have been volatilized from Type 1 than from the other sludge types because of the longer period of exposure. Type 1 sludge was expected to have a higher concentration of NH_4-N and NO_3-N than the younger Types 2 and 3 sludge. This was however, not the case as the concentrations of NH_4-N and NO_3-N was higher in Type 3 sludge which is the youngest of the three sludge types. Studies carried out by Wong *et al.* (2001) showed that during anaerobic digestion of sludge, most of the N is converted to NH_4-N which may justify its higher concentration in Type 3 sludge. In addition, NH_4-N is rapidly volatized on mineralization while most of the NO_3-N is easily leached out (Gilmour and Skinner 1999). These may therefore account for the lower values of NH_4-N and NO_3-N in the older Types 1 and 2 sludge than in Type 3 sludge.

Heavy metals concentrations in sludge: Both the values for Pseudo-total concentrations and the concentration of the leachable fraction of all metals analyzed were within the maximum acceptable concentrations stated by the Government of Botswana (2003), and the Permissible Utilization and Disposal of Sewage Sludge Edition 1, 1997 in South Africa (Snyman 2000) for sludge to be applied to soil. Heavy metal concentration in sludge is determined by the degree to which industrial wastewater has been mixed with the treated wastewater (European Commission, 2001). The relatively low level of industrialization in Botswana may explain the low values obtained for the pseudo-total concentrations of the different heavy metals in the three sludge types. Among all the metals analyzed, values for both the pseudo-total concentration and the concentration of potentially leachable fraction of Zn were the highest in all three sludge types, whereas Cd had the lowest values (Table 2). Arsenic and Cd were not detected in the leachable fraction of all three sludge types. These metals are usually associated with industrial activities which are not common in Gaborone city thus explaining their low concentrations in the sludge. Not being able to detect these heavy metals in the leachable fraction of these sludge types could be indicative of the low risk posed by these sludge with regards to the addition of Cd and As in the environment

Table 2: Concentration of Heavy metal in the different sludge types

Heavy metals	Concentrations in mg/Kg					
	Type 1		Type 2		Type 3	
	leachable	Pseudo-total	leachable	Pseudo-total	leachable	Pseudo-total
As	nd	3.9	nd	11.3	nd	14.8
Cd	nd	1.1	nd	1.5	nd	1.3
Cr	0.5	4.9	0.5	6.5	1.0	4.8
Cr	1.1	4.4	0.9	5.8	1.3	5.3
Cu	2.4	110.8	1.6	115.9	0.8	63.2
Mn	10.2	251.1	10.9	270.3	10.3	114.1
Ni	5.3	17.6	6.1	22.8	6	12.9
Pb	7.9	228.6	7.2	295.5	6.9	209.7
Zn	125.0	341.0	172.0	400.0	90.7	346.6

nd = not detected

Less than 40 % of the concentration of each heavy metal analyzed was leachable (Figure 1). Binding of heavy metals by OM contained in sludge (McBride *et al.*1997) may explain the low leachability of the heavy metals in Type 1 sludge. This is further justified by the scores for metal leachability which were 2.1, 1.9 and 2.3 for Types 1, 2, and 3 sludge respectively. Heavy metals adsorbed onto OM are desorbed on mineralization of the OM, which may explain the higher concentrations of leachable fraction of heavy metals in Type 2 sludge. The desorbed heavy metals are susceptible to leaching, to which the lower values for the concentrations of leachable fraction of heavy metals in Type 1 sludge could be attributed. Apart from Ni which preceded Zn in the leachability in Type 3 sludge, potential leachability of heavy metals in the three types of sludge followed the order Zn > Ni > Cr > Co > Mn > Pb > Cu (Fig 1).

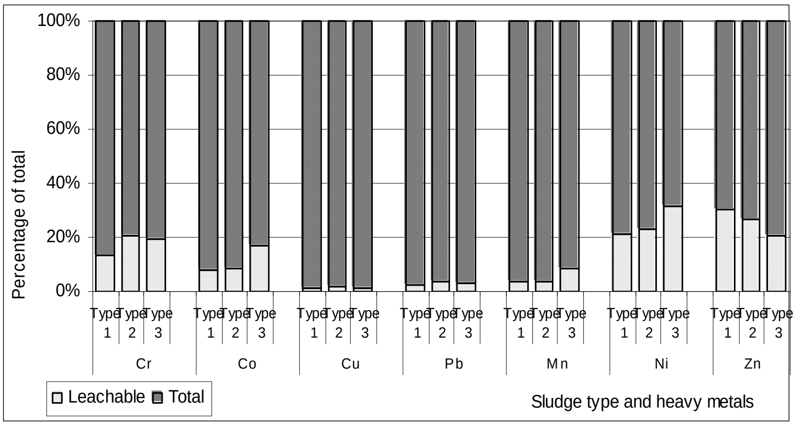

Fig 1: Distribution of heavy metal concentrations in the different sludge types between leachable and total fractions

Total and faecal coliform population in sludge: The suitability of faecal coliform as an indicator for faecal contamination has been documented by Windfield and Groisman (2003). Though TC are present in most environmental substrates, FC inhabits mostly the lower intestine of warm-blooded animals which justify their presence in the three sludge types. The population of both total coliforms (TC) and faecal coliforms (FC) in Type 3 sludge was higher than that in Type 2 sludge which was itself higher than the population in Type 1 sludge (Figure 2). These results indicated a decrease in coliform population with increase in the age of the sludge. Outside the host environment, coliforms are exposed to environmental factors such as temperature and moisture fluctuations, and the disinfecting and desiccating effects of the sun which all have a negative effect on their survival.

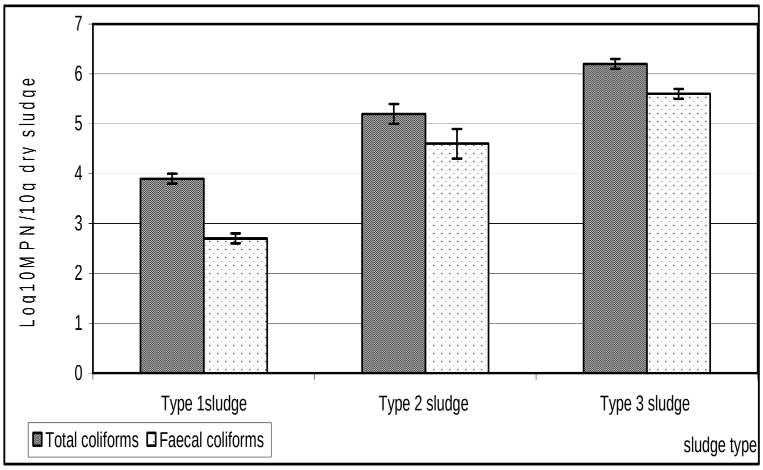

Fig 2: Total and faecal coliforms contained ion the three sludge types

A longer period of exposure to these factors as was the case with Type 1 sludge compared to Types 2 and 3 sludge explains the decrease in coliform population with increase in age of sludge. According to Epstein (1998) faecal coliform survival period in soil ranges from 12 – 268 days. The numbers indicated especially in Type 1 sludge which was over 3 years old could have resulted either from recontamination from sludge dust and leachate or regrowth of these organisms or both. Enterobacteria identified using API 20 E analyses included *Enterobacter cloaca* and *Enterobacter sakazakii* in Type 1 sludge, *Klebsiella pneumoniae*, and *Escherichia coli I (an API- group differentiated on the basis of biochemical reactions),* in the Type 2 sludge, and *Klebsiella pneumoniae, Escherichia coli I, Enterobacter cloaca*, *Enterobacter sakazakii*, *Yersinia intermedia*, and *Enterobacteria aerogenes* in Type 3 sludge.

Agronomic value of the three sludge types: Plant macronutrients contained in all three sludge types were comparable with those of other commonly used animal manures but the K content as expected of sludge (Brady and Weil, 1998) was lower (Table 3). The advantage of using sludge over these organic manures stems from the fact that it has a slower rate of organic nitrogen mineralization (Gilmour and Skinner, 1999). Nitrogen contained in sludge could therefore be available over a longer period of time thus reducing the frequency of application and potential risk of surface and groundwater contamination. In addition sludge contains Fe, Al, and Ca, which form insoluble phosphates (Maguire *et al.*, 2001). The risk of surface and groundwater contamination with P from sludge-amended soils is lower than from soils amended with other organic manures.

Table 3: A comparison of the nitrogen, phosphorus, and potassium values of various organic manures with that of Types 1, 2 and 3 sludge.

Kind of Manure	Nitrogen (%)	Phosphorus (%)	Potassium (%)
Type 1 sludge	3.4	0.7	0.5
Type 2 sludge	4.5	0.8	0.7
Type 3 sludge	5.5	0.9	0.6
Poultry manure*	4.4	2.1	2.6
Dairy cow*	2.4	0.7	2.1
Horse*	1.4	0.4	1.0
Sheep/goat*	3.5	0.6	1.0

**Source: Brady and Weil, (1998)*

Scores obtained from the plant nutrients contents and other parameters like SC, pH, EC and CEC that may affect sludge fertilizer value indicated that Type 1 sludge had a higher fertilizer value than Types 1 and 2 sludge (Figure 3). However, with the values for exchangeable sodium percent (ESP) being 21.5 %, 15.2 % and 10.1 % for Type 1, Type 2 and Type 3 sludge respectively, salinity problems may be encountered especially if Type 1 sludge is used. With regards to the VS and OM contents as well as the population of coliforms, Type 1 sludge had a better fertilizer value than the other sludge types (Fig 3).

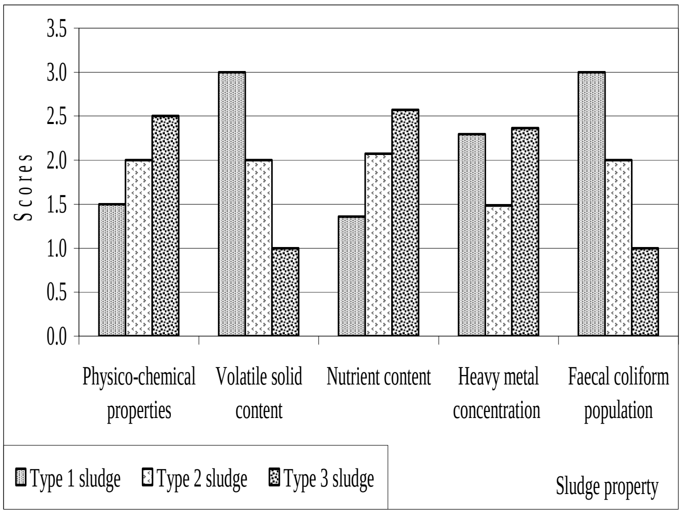

Fig 3: Fertilizer value of the three sludge types with regards to their constituents

If a considerable time is allowed between when the sludge is applied to the soil and when seeds are sown, the fertilizer value of the sludge may be improved. A previous study by Ngole et al., (2006) recommended that a minimum period of 90 days be allowed between when sludge is applied to soil and when seeds are sown in the soil to allow for further pathogen reduction. The specific period is however determined by the soil type on which the sludge is applied (Ngole et al, 2006).

The potentially leachable fraction of heavy metals contained in the sludge indicated the fertilizer value to be Type 3 > Type 1 sludge > Type 2 sludge with scores of 2.3, 2.1 and 1.9 respectively. However, the pseudo-total concentrations indicated a similar value for Types 1 and Types 3 sludge with Type 2 sludge being of lower value (Figure 3). The fertilizer value of the three sludge Types followed the order Type 1 sludge > Type 2 > Type 3 sludge with mean scores of 2.2, 1.9 and 1.9 respectively. Though Type 2 sludge had the same mean score as Type 3 sludge, Type 2 sludge may be considered to be of better value because of its score for faecal coliform population.

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