



# CHARACTERIZATION OF THE PHYSICO-CHEMICAL PROPERTIES OF THE MASERU MUNICIPAL WASTEWATER SLUDGE FOR POTENTIAL APPLICATION IN AGRICULTURAL SOILS AS AN ORGANIC-MINERAL AND SOIL MODIFIER

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The indiscriminate land disposal of wastewater sludge in Lesotho poses an environmental and health concern since sludge typically contains a myriad of hazardous chemicals. This study characterised the physico-chemical properties of the sludge generated at the Ratjomose Wastewater Treatment Plant in Maseru, the capital city of Lesotho and its chemical safety for application on land as an organic fertilizer. The sludge was slightly acidic and had considerable amounts of macronutrients. The heavy metal concentrations are less than the maximum limits recommended by the Food and Agricultural Organization and the South African standards for sludge applied to agricultural land and are slightly higher than those recommended in the national industrial effluent discharge standards in Lesotho. Principal component analysis did not reveal any major differences between the different cells used in the treatment plant. Since the nutrients and chemical composition of this sludge match the guidelines for an organic-mineral soil conditioner, it can be concluded that this sludge is safe for use in agricultural soils with no expected negative effects.

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## Introduction

The sludge is a solid end-product of wastewater treatment, whose composition depends on the source and type of the wastewater influent entering the treatment plant as well as the treatment processes employed therein.<sup>1</sup> Wastewater sludge typically contains about 50 % organic matter and significant amounts of plant macronutrients that are generally not present in conventional chemical fertilizers.<sup>2</sup> These factors make the application of sludge as a fertilizer and soil conditioner in agriculture a more attractive option than the conventional disposal in landfills.<sup>3</sup> The application of sludge to agricultural land ranges from as low as 0.06 % to as high as 70 % of the total sludge generated.<sup>4,5</sup> Primarily, sludge application to the land recycles the essential plant nutrients and organic matter into the soil, which improves the productivity of the soil and increases crop yields with yield enhancement reportedly as high as 20 %.<sup>6,7</sup> It improves the physical and chemical properties of soils; such as the organic matter content and electrical conductivity of soils that increase considerably,<sup>8</sup> lowered erosion rates, even before vegetation covers the soil surface,<sup>9</sup> the bulk density decreases, aggregate stability increases, all of which result in increasing water retention, aeration and permeability of some soils,<sup>10,11</sup> especially when the sludge is applied in the form of dewatered sludge cake.<sup>12</sup>

The other advantage includes the reduction in the use and costs of synthetic chemical fertilizers with savings as high as 40 % being reported when inorganic chemical fertilizers are

supplemented with wastewater sludge.<sup>7</sup> It has also been argued that the recycling of organic matter through the soil-plant system in the form of sludge, compost and manure will reduce the amount of carbon in the atmosphere.<sup>13</sup> Generally, municipal wastewater sludge contains toxic pollutants both of chemical and biological origin.<sup>14,15</sup> The bioavailability of sludge-borne metals in soils is influenced by the physical and chemical properties of the soil of which the pH, the cation exchange capacity and the organic matter content become the major influential factors over the long-term.<sup>16,17</sup> This therefore emphasizes the need for comprehensive characterization of sludge to mitigate the potential environmental and health impacts such as the potential risk of accumulation of toxic metals<sup>18,19</sup> and some high parasite concentrations<sup>20</sup> in soil and hence a necessity to determine the appropriate loading rates and application frequency in order to protect farmers and farm workers, soils, crops and the environment<sup>21,22</sup>. In view of these potential risks, application of sewage sludge to agricultural land is not accepted in many countries due to heightened public awareness and perception of risk as direct consequences of sludge application on land.<sup>23</sup>

In Lesotho, as in most other developing countries, sanitation focuses largely on the treatment of the wastewater and little attention is given to the management of the sludge generated, due to the high cost of treatment and disposal.<sup>4</sup> Sludge treatment, handling and disposal may be up to 50 % of the total costs for wastewater treatment. A large proportion of the sludge produced in Maseru, the capital of Lesotho, is generated at the Ratjomose Wastewater Treatment Plant (RWTP), the largest sewage treatment plant in Lesotho, capable of handling of 20 million cubic meters of wastewater per day. The municipal wastewater is treated using a combination of conventional and waste stabilization ponds methods. However, the guidelines for the management of the sludge generated are still at the drafting stage. Consequently, a large proportion of sludge generated

at wastewater treatment plants is disposed of indiscriminately in the abandoned quarries or recycled into agricultural land by individual subsistence farmers to improve soil fertility for crop production.<sup>24</sup> The current status favours the wastewater treatment company, because it is relieved of the burden and cost of further treatment and disposal of the sludge.

This study examined the physical and chemical characteristics of municipal wastewater sludge generated at the RWTP for its suitability for application on agricultural land, and compared the concentrations of nutrients and some common pollutants with South African standards and the FAO guidelines.<sup>25</sup> The data will also help to avoid the practice in most developing countries where effluent and sludge disposal standards are either too stringent because they are based on standards from developed countries or too relaxed so as to attract foreign industrial capital and therefore they do not guarantee the safety of the intended use of water and reuse of sludge in agriculture.<sup>26</sup>

## Experimental

The selection of parameters for the study was guided by either the beneficial or detrimental effects on soil and on plant growth, potential environmental and health impacts.<sup>21,27</sup> The parameters selected for determination are pH, electrical conductivity, organic matter, macronutrients – nitrates and phosphates, calcium, potassium, magnesium and sodium; micronutrients - iron, manganese, copper and zinc and seven potential toxic metals arsenic, cadmium, chromium, cobalt, lead, mercury and nickel.

### Sample collection, preparation and analyses

Sludge samples were collected from the RWTP and stored in the refrigerator at 4 °C till further use. Prior to analysis, the samples were air-dried at room temperature to a constant mass. Portions of the air-dried sludge were crushed and sieved through a 2-mm mesh to restrict particle size. These fractions were used to determine the physical and chemical parameters. To determine electrical conductivity (EC) and pH the sludge samples were blended in water (1:10 w/w) at 25 °C. The pH was measured using pH meter InoLab pH Level-1 (Germany), while conductivity was measured with conductivity meter, Condi-330i (Germany).

For other determinations, the representative samples were weighed and dried to a constant weight at 80±5 °C. A portion of the air-dried sludge samples was ground and sieved through a 1.0-mm mesh. This fraction was used to determine the total organic matter, nutrients, and the total content of potential toxic metals. Total organic carbon (TOC) was determined according to literature.<sup>28</sup> Organic matter (OM) was determined by wet digestion method. Nitrate-nitrogen and total phosphorous were determined according to the Hach methods 8039 and 8187.<sup>29</sup>

Solutions for the determination of metals were prepared by acid digestion of the sludge samples according to US-EPA Method 3050B.<sup>21</sup> Metals bound to organic carbon were released with 30 % H<sub>2</sub>O<sub>2</sub> and HNO<sub>3</sub> at pH 2.0 and 85 °C.<sup>30</sup> Sodium, potassium, lead and zinc were determined by AAS

FS-220 flame atomic absorption spectrophotometer (Varian, California, USA). The other metals were determined using LIBERTY AX Sequential ICP-AES spectrophotometer (Varian, California, USA). Standard solutions for the analysis were prepared according to the Varian AAS 1998 Handbook. All chemicals used were of analytical reagent grade (Merck and BDH) and all solutions were prepared using de-ionized water.

Multi-variate statistics, specifically, principal component analysis was carried out using MINITAB Version 14 for the inter-cell variation as well as analysis of the differences between the analytes.

## Results and Discussion

### Nutrient content of the sludge

The results for the nutrient content and some physical properties of the sludge are shown in Table 1. The sludge is moderately acidic, the pH values of all the samples are within the range from 5.45 to 7.25 with a mean pH of 6.05. Sludge pH is very crucial in determining the rates of application on land as it has a direct effect on the pH of the soil and the bioavailability of micronutrients and metal ions to plants.<sup>31,32</sup> An increase in sludge acidity could result in increase in the solubility of most metal ions like Pb, Cr, Cd, Cu, Ni and Zn and hence their bioavailability and uptake by plants.<sup>33</sup> The pH range for most agricultural practices is 6.5 to 7.5. Within this pH range most metal ions are bound to soil particles and therefore not accessible to plants.<sup>34</sup> In the untreated form, the sludge from the RWTP is only suitable for application to alkaline soils so as to neutralize the alkalinity rather than as a mere bio-fertiliser.

**Table 1.** The major nutrient contents and some physical properties of the sludge samples.

Parameter	Cell 1	Cell 2	Cell 3	Cell 4
pH	6.01	5.45	5.50	7.25
Conductivity, $\mu\text{S cm}^{-1}$	703	784	776	277
Total organic matter, $\text{g kg}^{-1}$	520	350	548	554
Total organic carbon, $\text{g kg}^{-1}$	1660	1733	1656	2871
TDS, $\text{g kg}^{-1}$	566	522	554	770
Organic-nitrogen, $\text{g kg}^{-1}$	15.8	19.2	13.6	47.4
Nitrate-nitrogen, $\text{g kg}^{-1}$	32.6	26.4	38.2	51.6
Total N, $\text{g kg}^{-1}$	48.4	45.6	21.8	99.0
Total P, $\text{g kg}^{-1}$	24.5	5.7	28.5	13.4
Ca, $\text{g kg}^{-1}$	31.5	35.2	25.3	24.7
Mg, $\text{g kg}^{-1}$	6.2	9.4	8.3	5.3
K, $\text{g kg}^{-1}$	1.8	4.3	2.6	2.4
Na, $\text{g kg}^{-1}$	3.2	4.5	2.1	1.8

EC is a surrogate measure of total dissolved solids (TDS) and also has a direct correlation with salinity, which is the total concentration of dissolved ionic salts in the soil solution. Therefore, it is expected that the total dissolved solids would correlate positively with the electrical conductivity, but the relationship is not direct because the mobility of ionic species in solution is variable and depends

on the nature and type of ions in the solution. The EC value for the fourth drying cell is half its value for TDS, which indicate that the dissolved solids were both ionic and non-ionic in nature. Salinity is an important parameter of soil and high salinity has toxic effects.<sup>35</sup> Thus the application of sludge with high levels of salts could ultimately affect the growth of plants.<sup>36</sup>

The organic matter content of the sludge is in the range 350 to 554 g kg<sup>-1</sup> of dry weight, which translates to 35.0 to 55.4 %, which is typical of municipal wastewater sludge.<sup>37</sup> The overall effect of the changes brought by the sludge could be the reduced amounts of runoff and soil erosion,<sup>6</sup> which will be very beneficial to farmers in Lesotho where soil erosion is major problem.

Regarding the major nutrients shown in Table 1, the total nitrogen content, determined as the sum of the organic and inorganic nitrogen, fell in the range 2.2-9.9 % while total phosphorous fell within 0.6-2.9 %. These values compare very well with those of some common organic manure and provide similar fertilizing properties (see Table 2). Nitrogen is an integral component of chlorophyll and therefore essential for photosynthesis and is important in periods of rapid plant growth. It is also a vital component of proteins, which control the metabolic processes required for plant growth. An adequate supply of nitrogen is associated with vigorous vegetative growth and a plant's dark green colour. Nitrogen deficiency is characterized by reduced plant growth and a pale green or yellow colour.<sup>38</sup>

Phosphorus is a critical component of nucleic acids and plays a vital role in plant reproduction and it is essential to seed formation. Phosphorus is essential for the biological energy transfer processes that are vital to life and growth<sup>39</sup>. Phosphorus deficiency is indicated by reduced plant growth, delayed maturity, and small fruit set. The availability of phosphorous is very sensitive to the soil pH with the uptake rate by higher plants being at the highest between pH 5.0 and 6.0 where H<sub>2</sub>PO<sub>4</sub><sup>-</sup> predominates in soil solution.<sup>40</sup> The sludge contains significantly higher amounts of total nitrogen than that in cattle, poultry and sheep manures (Table 2). The phosphate content is higher than that of cattle and sheep manure, but lower than phosphate content of poultry manure.

The concentrations of exchangeable bases are in the range of 1.80-9.40 g kg<sup>-1</sup> of the dried mass. The lowest value was recorded for Na<sup>+</sup> while the highest value was for Ca<sup>2+</sup>. These values are much higher than the concentrations determined for cattle and sheep manures (Table 2), but are lower than the concentrations in poultry manure. However, the nutrient contents are lower than those of synthetic inorganic fertilizers on the market. Most nitrogenous fertilizers contain 15-40 % total nitrogen, phosphate fertilizers contain 14-30 % of phosphates, which may be in the water soluble form or citrate soluble form and potash fertilizers contain 22-60 % of water soluble potash.<sup>41</sup> Though the nutrients contents are low, relative to chemical fertilizers, the sludge can be a good alternative as a soil conditioner because land application reduces or eliminates the cost of disposal and use of synthetic mineral fertilizers.

The comparison of the values from cell to cell did not seem to follow any trend as can be seen from Figure 1. Some parameters showed a decrease from cell 1 to 4, some do not show any trend.

**Table 2.** Comparison of nutrients content of used sludge sample and some common organic manure.

Manure	Average nutrient content (% by weight)				
	Total-N	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	CaO	MgO
Poultry*	2.20	1.80	1.10	2.40	0.70
Cattle*	1.20	0.17	0.11	0.35	0.13
Sheep*	1.55	0.31	0.15	0.46	0.15
Maseru	8.50	1.20	0.34	3.01	0.75
Sludge		(PO <sub>4</sub> <sup>3-</sup> )	(K)	(Ca)	(Mg)

\*Source: SRI-CSIR, 1997 Soil Research Institute (SRI). 1998. The 1997 Annual Report. Accra.

### Heavy metal content

The presence of toxic heavy metals in sludge makes its use as an organic fertilizer or soil amendment a matter of concern. Furthermore, the uptake of these metals by plants and their subsequent bioaccumulation along the food chain poses a potential threat human health. The total concentrations of the heavy metals in the sludge are shown in Table 3.

**Table 3.** Metal contents of sewage sludge from Ratjomose Wastewater Treatment Plant.

Metal	Total conc. mg kg <sup>-1</sup>	Bound to organic carbon,		Maximum limit*, mg L <sup>-1</sup>
		mg kg <sup>-1</sup>	%	
Al	37.5	21.4	57.1	NA
As	13.5	8.50	65.4	0.5
Cd	8.5	7.34	86.4	0.05
Co	172.2	29.3	17.0	NA
Cr (total)	7.4	4.31	58.2	0.5
Cu	85.0	65.3	76.8	1.0
Fe	1424	1121	78.7	NA
Hg	4.2	3.76	89.5	0.02
Ni	18.0	8.60	47.8	NA
Pb	25.8	0.8	3.1	0.1
Zn	4438	843.6	19.7	5.0

\*National Environment Secretariat, Wastewater Discharge Standards, First Draft, Maseru, 1997; NA – not included in draft standards.

The higher concentrations of these heavy metals in the sludge could only originate from the textile industries, since there are no other chemical industries in Maseru. These industries discharge their effluents into the sewer systems with minimum pre-treatment.<sup>41</sup> Textile industries effluents contain heavy metal ions from metal containing dyes.<sup>42,43</sup> In general all the sludge samples contain relatively higher amounts of all the target metals.

It was observed that the sludge sample with the higher organic matter content has a higher proportion of the heavy metals bound to organic carbon. Most of the metals have a high proportion bound to organic matter in a decreasing order: Hg (89.5 %) > Cd (86.4 %) > Cu (76.8 %) > As (65.4 %) > Cr (58.2 %) > Ni (47.8 %) > Zn (19 %) > Co (17 %) > Pb (3.1 %).

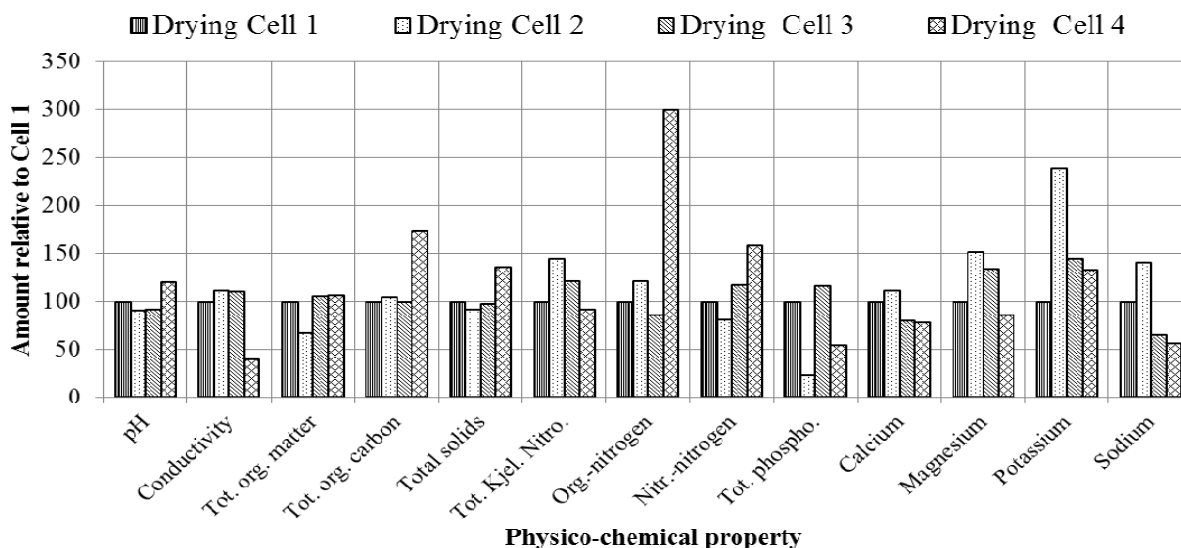


Figure 1. Some physico-chemical properties of sludge of in different cells relative to those in cell 1.

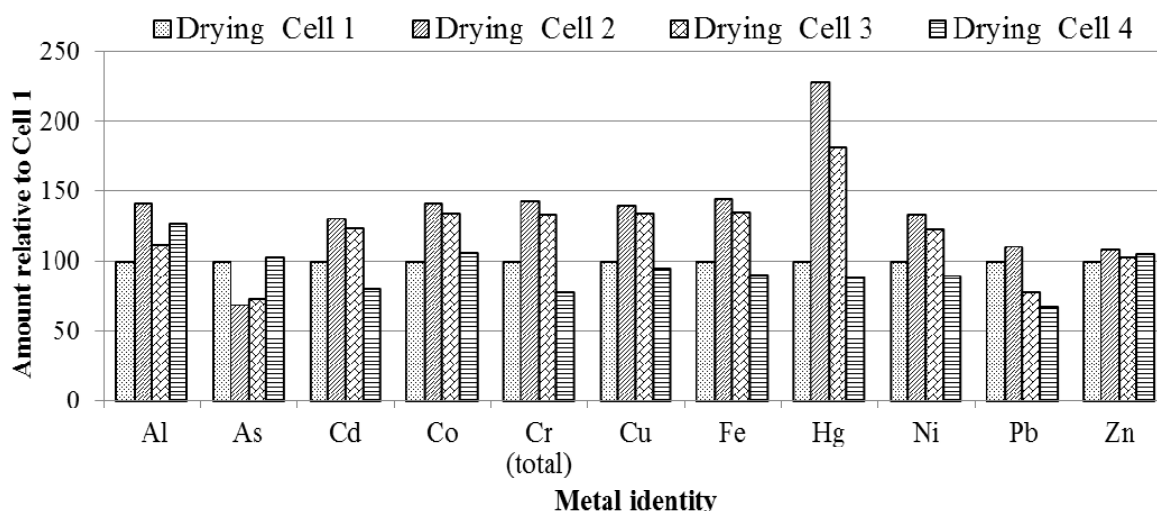


Figure 2. Relative abundances of some heavy metals measured relative to cell 1.

This suggests that these metals may have formed stable insoluble complexes with the organic fraction or they are strongly bound to the organic matter, limiting their solubility and potential bioavailability in soil.<sup>15</sup> Depending on the pH of the soil, these organic-bound metals may be slowly released and become bioavailable to plants.<sup>44</sup> This assertion agrees with the slight increases in metal concentrations with increasing acidity of the sludge. All the sludge samples exhibited elevated levels of the metals, higher than the maximum limits set out in the draft wastewater and industrial effluent discharge standards for Lesotho.<sup>45</sup>

Interestingly, it was observed that the concentrations of the metals increase slightly with the increasing acidity, i.e. decreasing pH of the sludge.

Figure 3 is a plot of relative abundances of individual ions (relative to pH 7.25, near neutral measured pH) against the actual pH of the media.

The metal concentrations were compared with the South African sludge standards and the FAO recommended guidelines for sludge application on land, as shown in Table 4 because there are no national standards in Lesotho. In all the sludge samples, only the concentration zinc ( $4438 \text{ mg kg}^{-1}$  dry weight) exceeded the maximum permissible value of  $3000 \text{ mg kg}^{-1}$  dry weight of the South African standards, but lower than the  $7500 \text{ mg kg}^{-1}$  weight recommended by FAO. Heavy metals are a concern because they are non-biodegradable and over time they accumulate in the plough layer of the soil, where they are available to plants.



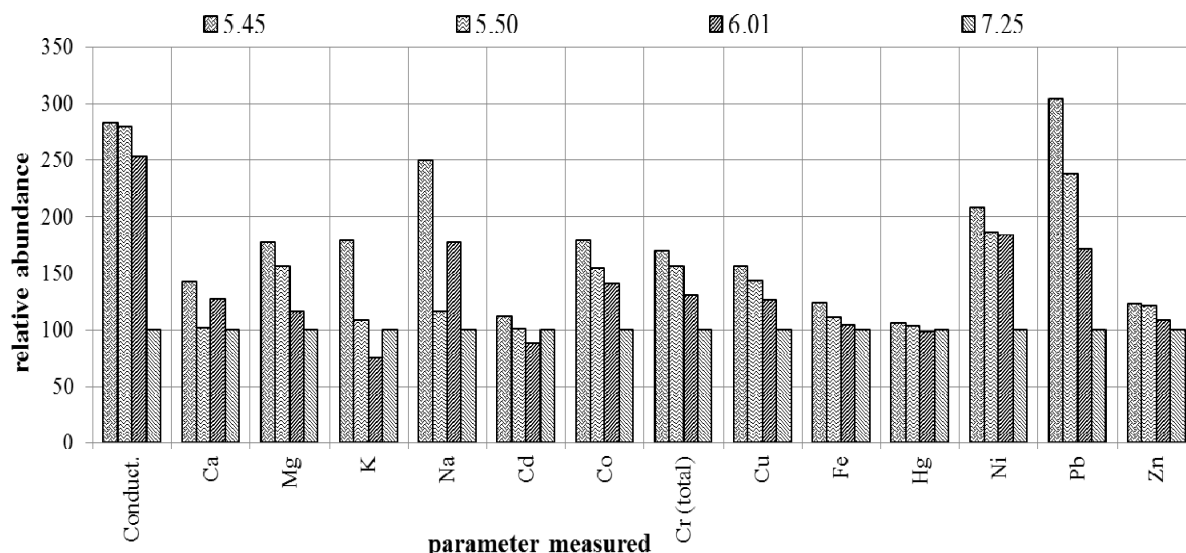


Figure 3. The pH dependence of the abundance of the metal ions from the sludge relative to pH 7.25.

Table 4. Comparison of heavy metals concentration in sewage sludge with international standards.

Metal	Amount, mg kg <sup>-1</sup> (dry weight)		
	RWTP Sludge	South African Standards	FAO Guidelines
As	13.0	80	75
Cd	18.5	20	85
Co	56.4	-	-
Cr	7.4	1200	3000
Cu	85.0	1200	4300
Hg	4.2	25	57
Ni	18.0	200	420
Pb	25.8	1200	840
Zn	4438	3000	7500

Most heavy metals such as cadmium, build up in the soil with continued sludge applications can be toxic to soil micro-organisms and thus affecting soil fertility and physical structure.

The mean concentrations of the metals in the sludge samples are shown in Table 2. There is no general agreement concerning the maximum allowable concentrations of various metals in sewage sludge. The concentrations of all the metals are several times lower than the recommended maximum permissible contaminant levels (MCL) set by most regulations bodies. For example, the South African Standards (SAS) set the MCL for zinc at 4438 mg kg<sup>-1</sup> dry sludge while the US EPA set the value at 7500 mg kg<sup>-1</sup> dry sludge; both significantly higher than the obtained values of 3000 mg kg<sup>-1</sup>.

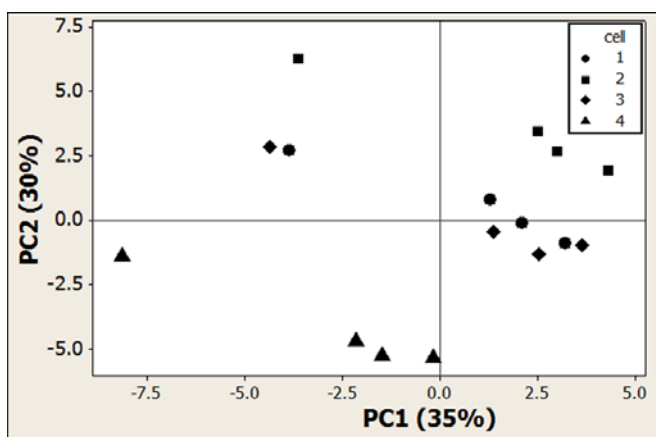
With this comparison, it can be concluded that the sludge can be applied safely to the agricultural land. However, MCL values vary with the pH of the soil because it is known

that crop damage from phytotoxic elements is more likely to occur on acid soils. The MCL values for Zn are 200 mg kg<sup>-1</sup> dry sludge at pH 5.0-5.5; 250 mg kg<sup>-1</sup> dry sludge at pH 5.5-6.0; 300 mg kg<sup>-1</sup> dry sludge at pH 6.0-7.0 and 450 mg kg<sup>-1</sup> dry sludge at pH >7.0 (see Table 3). Thus, the pH of the sludge is very vital when deciding on the rate of application of sludge on land. The phytotoxicity from the sludge metals usually is not clearly apparent for most plants as long as the soil pH is maintained in the 6-7 range by lime amendment.

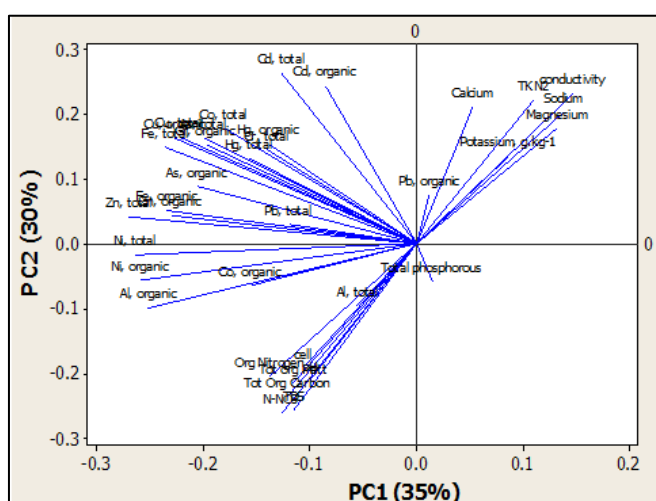
**Analysis of the data with multivariate statistics**

Multi-variate statistics is usually used to reveal some valuable information that may be hidden from the human eye at the same time reduce the complexity of the data to only few dimensions that could be easily identified. This technique was successfully applied recently in the profiling of the Caledon River along Maseru City where it demonstrated major contributors to the general water pollution in this important source of potable water to the municipality other than being an international border with South Africa, the only neighbour to Lesotho.<sup>45</sup> Figure 4 shows the scatter plot following the analysis of the data by principal component analysis.

As can be seen, the data shows that there is no considerable difference between the cells (PC1) but rather a larger difference occurred in between the trials (still along the PC1), a situation that is quite strange. This possibly implies that the samples may not have been homogenous enough to yield similar and comparable replicate results. This assertion is supported by the loadings plot (Figure 5) which reveals that a majority of these parameters are responsible for the shift in the PC1, while PC2 corresponds to the variation between the individual cells, which is usually the opposite. However Cell 4 seems to be the one that is slightly different from the rest of the cells with about 5 % variance (Figure 4) while Cells 1 and 3 are almost replicates.



**Figure 4.** A scatterplot showing the variance between the cells and the replicates



**Figure 5.** A loadings plot showing individual parameters that were measured in the study

The small difference observed between the cells was to be expected since the cells are used interchangeably, where the next cell is used when the previous gets emptied, and so on. The shift in the PC2 due to nitrogen content is clearly expressed in Figure 1 for Cell 4. This has thus rendered Cell 4 different from the other cells. However, overall, this difference in the cell is quite negligible. These results further demonstrate the applicability of multi-variate statistics in reducing the multiplicity of data into something manageable to screen for major contributors to the observed trends.

## Conclusions

The sewage sludge produced at the Ratjomose Treatment Plant has been characterized for its physico-chemical properties. The results showed that the sludge can be a good source of organic matter, plant macro- and micro-nutrients. It has higher nutrient content than cattle, sheep and poultry manures, although these contents are still lower than the conventional NPK fertilizers. The pH varies in a very narrow range of pH 6.4 - 7.6, which is close to neutrality and ideal for most soils. However, the sludge contains some toxic heavy metals, but the levels are much lower than the

guideline values recommended by FAO, but higher than the recommended in the draft national standards. A high proportion of these metals is bound to organic carbon in the order: Hg (89.5 %) > Cd (86.4 %) > Cu (76.8 %) > As (65.4 %) > Cr (58.2 %) > Ni (47.8 %) > Zn (19 %) > Co (17 %) > Pb (3.1 %), and hence may not be readily available for plant uptake. There seems to be a direct relationship between high acidity and the release of these heavy metal ions from the organic matter. Besides the intra-trial differences, something considered a possibility of batch error; the four cells seem to have uniform compositions. It can thus be concluded that this sludge can be applied to agricultural soils with confidence as it contains very low levels of hazardous components. The presence of hazardous heavy metals, albeit at considerably low levels, requires some thought if this sludge would have to be regularly applied to the same field to avoid eventual accumulation of these ions.

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