Full Length Research Paper

Chemical characterization to evaluate the agricultural potential use of organic wastes generated by industrial and urban activity

Cácio Luiz Boechat[1]* and Poliana dos Santos Pereira da Silva[2]

[1]Soil Department, Federal University of Rio Grande do Sul, Avenue Bento Gonçalves, 7712, Postal code 91540-000, Porto Alegre city, RS state, Brazil.
[2]Soil Department, Federal University of Recôncavo da Bahia, Rui Barbosa street, 710, Postal code 44380-000, Cruz das Almas city, BA state, Brazil.

The use of organic waste as a source of plant nutrition and/or to minimize problems in agricultural soils is one possible use of these materials. The objective of this study was to evaluate the characteristics and some chemical properties of organic wastes from different processes and sources to know the potential for agricultural use and the risks associated with their use. Data were subjected to an analysis of variance (ANOVA) to examine the chemical differences between organic wastes with three replications. A statistical analysis was conducted using the software SAS. The comparison of means was performed using the Turkey's test at the significance level of ($P < 0.01$), with three replications. The wastes were separated into acidic, neutral and basic classes. The Ca, Mg, P and N were characterized as an excellent source of these elements in organic and inorganic form, but the C/N ratio limited the use of some of these wastes according to the release time of nutrients expected. Micronutrients and heavy metals are below maximum allowable concentrations by environmental agencies and government.

Key words: Organic sludge, pulp mill sludge, sewage sludge, C/N ratio, trace elements.

INTRODUCTION

According to the European Commission for the period of 2003 to 2006, about 10 million tons in dry matter (DM) of sewage sludge were produced in the European Union (EU); 8.7 million t DM in the EU-15 and an additional 1.2 million t DM for the 12 new member states. No data was reported for Malta (European Commission, 2010). In Brazil, in 2008 and 2009, approximately 46,550,088 and 50,258,208 tons of urban solid organic waste (sludge), were collected, of these, 57 and 55% respectively were disposed of appropriately (ABRELPE, 2009). The use of organic wastes from production processes as a source of plant nutrition in agricultural soils is one of the ways to employ these materials, as they increase fertility, and improve soil characteristics and physicochemical

properties, and also microbial activity (Brady and Weil, 2002). When this organic waste is applied, the nutrients must be strictly monitored. There have been many studies to investigate the effect of organic wastes on the physic-chemical characteristics of soil (Abu-zreig and Al-Widyan, 2002), their biological properties (Brady and Weil, 2002; Fioretti et al., 2001) crop yields (Cooperband et al., 2003) and vermin composting (Dores-Silva et al., 2011).

The waste from industrial or urban areas can be a source of heavy metals. This causes great concern regarding their use to fertilize plants that provide human food (Wang et al., 2003), as well as the possibility of groundwater contamination (Barros et al., 1995). The greater or lesser mobility of these metals in soil will depend on several intrinsic soil characteristics especially, pH (Amaral et al., 1992) and the number of adsorption sites (Sparks, 1995). Moreover, even in trace amounts,

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*Corresponding author. E-mail: clboechat@hotmail.com.

some of these elements are very toxic and as they become incorporated to the soil, the effect may increase (Moura et al., 2006).

This study aimed to characterize chemically organic wastes from different processes and sources for the levels of organic carbon, total-N, mineral-N, Ca, Mg, P, Fe, Cu, Mn, Ni, Cd, Pb, Cr, and pH_{H2O}, pH_{CaCl2} and neutralizing capacity, thus, confirming the potential use of agricultural waste, either to correct soil acidity or as a source of plants nutrients and a potential risk of environmental or soil contamination by heavy metals.

MATERIALS AND METHODS

Experimental site, source and waste collection

The experiment was conducted in the laboratories of the Federal University of Recôncavo da Bahia and Embrapa Mandioca e Fruticultura, in Brazil, in 2010. The wastes were from different sources with distinct characteristics, namely: (RC) Treated pulp mill sludge - The raw material for this industry is newspaper, books, bank and office organic wastes and other paper that can be recycled. In this industry, the bio-reduction mass process is used to minimize the volume of solid waste from the Wastewater Treatment Plant; (RP) Petrochemical complex sludge - Sludge derived from organic wastes generated by industries located in the petrochemical complex and treated by wastewater treatment plant; (RM) Treated urban sewage sludge - This organic waste was the result of sewage treatment that used physical processes to remove coarse solids, biological activated sludge to remove biochemical oxygen demand, plus the addition of lime (CaO); (RL) Treated dairy industry sewage sludge - Organic waste generated by the dairy industry to manufacture cheese, and process butter and milk. The organic waste is aerated in a pond to reduce the organic load and eliminate pathogens, and chemical stabilization is performed applying lime (CaO); and (RF) Organic compost from the fruit pulp industry - derived from the remains of fruit peel and seeds for frozen fruit pulp production. The organic compost is piled for primary fermentation processes and partial decomposition of the material and stabilization of organic load for three years. All wastes collected were placed in the shade for partial loss of moisture. About 20 sub-samples of these were taken from each batch at 20 points, forming a compost sample of approximately 2000 g for chemical analysis. The samples were crushed to allow a homogeneous particle size and finally dried at 60 ± 2°C in a laboratory oven.

Chemical attribute of the wastes

In order to determine the neutralizing capacity, 150 g of soil were used, into which each waste studied was incorporated at the same rate based on the nitrogen content applied. The experimental units were placed in a BOD incubator in the absence of light, temperature controlled at 25 ± 0.2°C and humidity mai ntained close to 70% of field capacity. The moisture content was checked by weighing the experimental units and adjusted with deionized water. It was evaluated at 0, 2, 7, 14, 28, 42, 56, 70, 91 and 112 days of incubation with three replications. The chemical attributes analyzed were pH in water and $CaCl_2$. The pH was determined using a glass electrode by potentiometer, at a waste: deionized water or calcium chloride ratio of 1: 2.5, after shaking followed by 60 min of rest and stirring again during the reading (Quaggio and Raij, 2001). The total-N content was determined from samples previously dried at 60 ± 2°C and ground in a Wiley mill and sieved (0.25 m m). In this method, the product was weighed in digestion tubes, 0.100 g ± 1

mg of the product. Then, 1 g ± 1 mg of digesting mixture made by mixing 1 g ± 1 mg of K_2SO_4 + 100 g ± 1 mg of $CuSO_4.5H_2O$ + 10 g ± 1 mg selenium sieved in 0.5 mm (Mesh 35) were added, and after this, 3 ml of concentrated H_2SO_4 were added and placed in a block digestor covered with a small funnel (25 mm diameters) and heated gently until it reaches a temperature of 360 ± 5°C. T he mixture remained at a temperature of 360 ± 5°C for three hours until the tube was clear (Embrapa, 1999). To mineral-N (Bremmer and Keeney, 1966; cited by Tedesco et al., 1995), it is recommended to use 1.0 g ± 1 mg of dry sample, placed in 100 ml distillation tube and adding 15 ml of KCl 1 M to the material and shaking intermittently for five minutes. This was followed by distilling after the addition of 0.2 g ± 1 mg of calcinated MgO and collecting 35 to 40 ml of the distillate in five ml of boric acid indicator 20 mol L^{-1} (pH ± 5.0) and titrating it with H_2SO_4 0,0025 M for the content of NH_4^+. After the first distillation, 0.2 g ± 1 mg of fine Devarda's alloy was added (50% Cu, 45% Al and 5% Zn) and the distillation was started again in five ml of boric acid indicator 20 mol L^{-1} (± pH 5.0) performing new titrations, and obtaining a content of NO_2^- + NO_3^-. The concentrations of total-N and mineral-N were determined in distilled steam by a semi-micro Kjeldahl distiller.

The carbon content was determined by wet oxidation of organic carbon by $K_2Cr_2O_7$ in the presence of H_2SO_4 with external heating according to NBR 10357. Determination of organic carbon initially weighed 0.05 g of each waste in triplicate, and then moved the material to 500 ml Erlenmeyer flasks. This was added to precisely 10 ml of 0.067 M $K_2Cr_2O_7$ and then 20 ml of concentrated H_2SO_4. The bottle was immediately stirred to mix the waste with the reagents for about 1 min. The extract was left to stand for an approximate time of 40 min. Then, deionized water, 1 ml of H_3PO_4 and 1.0 ml of diphenylamine indicator 10 g L^{-1} were added, followed by titration with $[(NH_4)_2Fe(SO_4)_2.6H_2O]$ 0.102 M until the color changed from violet to green. At this point, the excess dichromate was completely consumed by the reaction. In parallel, the same procedure was done for the test blank. The same procedure was performed in parallel for the test blank.

Chemical analysis of elements

The Ca, Mg, P, Fe, Cu, Mn, Ni, Cr, Cd and Pb were determined. The method described by Embrapa12 was used to analyze the total content of these elements. Melo and Silva (2008) noted that nitric-perchloric digestion promotes the recovery of higher levels of nutrients in different organic wastes studied, compared to other procedures. This methodology started by weighing 0.50 g of the product previously passed through a sieve with a 0.15 mm mesh into digestion tubes of the product previously passed through a sieve with a mesh of 0.15 mm. This was followed by the addition of 6 ml of the mixture of nitric acid (HNO_3) with concentrated perchloric acid ($HClO_4$) (2:1) and the tubes were transferred to a block digester to be heated to a temperature of 50, 100 and 150 ± 5°C for 30 and 10 min to 200 ± 5°C. They were cooled , and 25 ml of deionized water added. Finally, they were stored in plastic containers (Embrapa, 1999). The concentrations of Ca, Mg, Fe, Cu, Mn, Ni, Cr, Cd and Pb were determined in an atomic absorption spectrophotometer (AA) Perkin - Elmer Analyst 100. An aliquot of 5 ml was withdrawn from the nitroperchloric extract, and diluted 30 times with deionized water. Then a 0.50 ml aliquot was withdrawn, and a reading was performed in the spectrophotometer, at a 420 nm wavelength by colorimetric (Embrapa, 1999).

Data analysis

Data were subjected to an analysis of variance (ANOVA) to examine the chemical differences in the organic wastes, with three replications. A statistical analysis was conducted using the software

Table 1. Chemical attributes of different types organic wastes, total value in dry base.

Chemical attribute[a]	Unit	Organic wastes[b]				
		RL	RM	RP	RC	RF
PH	Water (1: 2.5)	6.90[b]	5.67[c]	7.40[b]	8.30[a]	5.40[c]
P	(g dm^{-3})	15.00[a]	9.49[b]	4.04[c]	0.28[d]	0.51[d]
Organic carbon	(g kg^{-1})	161.60[b]	235.00[a]	34.40[c]	236.40[a]	232.40[a]
N- Ammonium	(mg kg^{-1})	6182.40[b]	8619.80[a]	750.12[c]	263.20[e]	460.60[d]
N- Nitrate	(mg kg^{-1})	36.96[d]	421.12[b]	855.40[a]	171.08[c]	881.72[a]
N- Kjeldahl	(g kg^{-1})	19.20[b]	32.63[a]	4.49[c]	3.72[c]	19.50[b]
C/N ratio	-	8.42[c]	7.20[c]	7.66[c]	63.55[a]	11.92[b]

[a] total value; [b] Mean value followed by the same letter do not differ significantly (Turkey's test, P<0.01); (RC) Treated pulp mill sludge; (RP) Petrochemical complex sludge; (RM) Treated urban sewage sludge; (RL) Treated dairy industry sewage sludge and (RF) Organic compost from the fruit pulp industry.
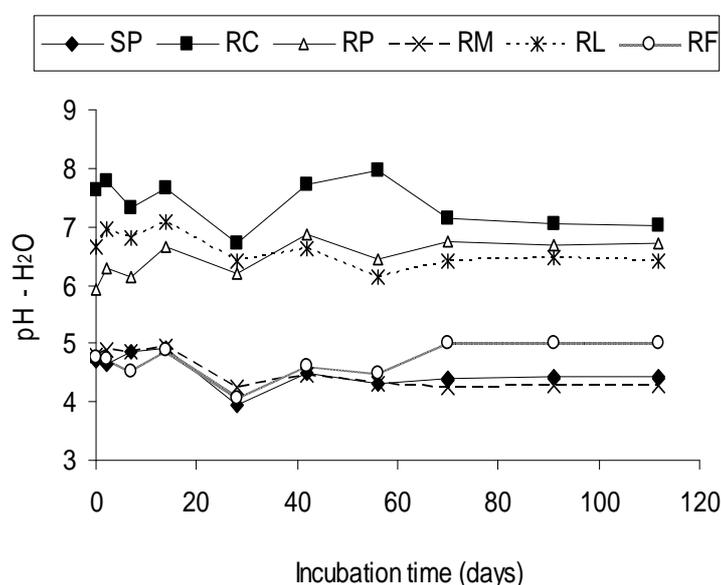
Figure 1. Influence of application of different types of waste in soil pH$_{H2O}$ over 112 days. (SP) Soil without waste; (RC) Treated pulp mill sludge; (RP) Petrochemical complex sludge; (RM) Treated urban sewage sludge; (RL) Treated dairy industry sewage sludge and (RF) Organic compost from the fruit pulp industry.

SAS (SAS Institute, 2004). The comparison of means was performed using the Turkey's test at the significance level of (P < 0.01) with three replications.

RESULTS AND DISCUSSION

Chemical attributes of the waste

The wastes were classified into three groups according to pH values. First - acid waste: RM and RF; Secondly - neutral waste: RL; and thirdly - alkaline waste: RP and RC, and shown to be related to the type of treatment to which the wastes are subjected in the production zone

(Table 1). Waste efficiency to change the soil pH was tested by incubation of the different wastes for 112 days (Figures 1 and 2). The alkaline and neutral wastes (RP, RL and RC) increased soil pH, while the RM and RF, similar to the control did not change soil pH. The pH of the treatments measured in water and calcium chloride was stabilized at 70 days of incubation. In this regard, the RP, RL and RC wastes were products that can be used to raise the pH of agricultural soils. On the first day RC, RP and RL treatments increased the pH of the soil due to calcium content in these organic wastes, which are 303.75, 121.25 and 178.75% respectively higher than the control (blank soil). The RM and RF treatments did not
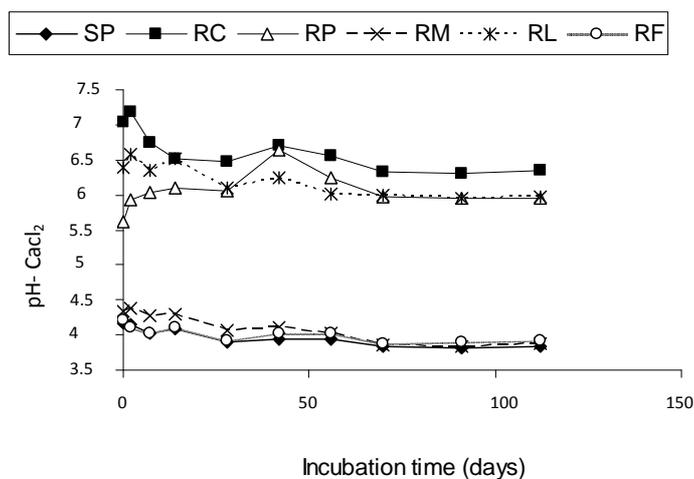
Figure 2. Influence of application of different types of waste in soil pH_{CaCl2} over 112 days. (SP) Soil without waste; (RC) Treated pulp mill sludge; (RP) Petrochemical complex sludge; (RM) Treated urban sewage sludge; (RL) Treated dairy industry sewage sludge and (RF) Organic compost from the fruit pulp industry.

Table 2. Chemical elements concentration in different types organic wastes, total value in dry base.

Chemical element[a]	Unit	Organic wastes[b]				
		RL	RM	RP	RC	RF
Ca	($cmol_{c\ dm}^{-3}$)	198.20[a]	59.86[d]	116.80[c]	190.23[b]	28.20[e]
Mg	($cmol_{c\ dm}^{-3}$)	13.05[c]	27.18[a]	5.48[d]	16.61[b]	18.50[b]
Cu	($mg\ kg^{-1}$)	141.79[b]	334.02[a]	373.20[a]	162.20[b]	101.72[b]
Fe	($mg\ kg^{-1}$)	11,752.69[a]	7,364.59[a]	7,640.86[b]	3,240.86[c]	701.08[d]
Mn	($mg\ kg^{-1}$)	292.28[a]	113.39[b]	83.58[c]	50.34[d]	68.43[c]
Ni	($mg\ kg^{-1}$)	1.13[a]	0.77[ab]	1.10[a]	0.70[ab]	0.27[b]
Cd	($mg\ kg^{-1}$)	0.12[a]	0.12[a]	0.12[a]	0.11[a]	0.10[b]
Pb	($mg\ kg^{-1}$)	44.44[a]	8.55[b]	32.48[a]	37.61[a]	3.42[b]
Cr	($mg\ kg^{-1}$)	6.83[a]	3.82[b]	4.222[b]	6.22[a]	0.60[c]

[a] total value; [b] Mean value followed by the same letter do not differ significantly (Turkey's test, P<0.01); (RC) Treated pulp mill sludge; (RP) Petrochemical complex sludge; (RM) Treated urban sewage sludge; (RL) Treated dairy industry sewage sludge and (RF) Organic compost from the fruit pulp industry.

show a changed capacity of soil pH due to their lower calcium and magnesium concentrations (Table 2).

The P and N concentrations were high in the wastes. However, the P content in RL was approximately 1.58, 3.71, 53.57 and 29.41 times higher than in RM, RP, RC and RF, respectively (Table 1). Although, it is an element used by plants at low concentrations when compared to the other nutrients, the addition of larger amounts of P to some Brazilian soils is recommended, due to adsorption on the surface of iron, aluminum oxides and hydroxides, and clay minerals which may be precipitated in the soil as salts of aluminum, iron and calcium with low solubility, making it unavailable to crops.

The RM had a content 1.70, 7.27, 8.77 and 1.67 times higher for the total-N content (Table 1) than RL, RP, RC and RF, respectively. N is the main element required by many crops. The nitrogen content in sewage sludge has been one of the parameters to calculate the amount to be applied, according to crop needs (CETESB, 1999). However, these two elements must be carefully observed at the time when they are applied from waste, because water resources have a high ability to be degraded by them. The P even at a moderate concentration in natural waters (25 μg/L), can stimulate the eutrophication process (Haygart and Jarvis, 1999; Hart et al., 2004) and N also may contribute to the eutrophication process, as it can contaminate groundwater with. This, when ingested by young animals or babies under six months cause

Table 3. Maximum permissible concentration in the sewage sludge or derived product based on dry mass.

Inorganic substances	CONAMA (2006)	MAPA (2006)	U. S. EPA (1993)
	mg kg^{-1}		
Arsenic	41.0	20.0	75.0
Barium	1,300.0	-------	------
Cadmium	39.0	3.0	85.0
Lead	300.0	150.0	840.0
Copper	1,500.0	-----	4,300.0
Chrome	1,000.0	200.0	------
Mercury	17.0	1.0	57.0
Molybdenum	50.0	-----	75.0
Nickel	420.0	70.0	420.0
Selenium	100.0	80.0	100.0
Zinc	2,800.0	----	7,500.0

poisoning or blue baby syndrome leading to death.

Organic carbon is also present at high concentrations in the wastes (Table 1). The content of this element in the product is directly related to organic matter content, which is associated with soils that have important chemical, physical and biological properties, such as water retention capacity, combination with clay minerals, chelation capacity, buffering capacity, increased cation exchange capacity and increased potential mineralization of essential elements for plants. It is a substrate for biological activity, improving the soil structure and soil aggregation capacity among others.

Another chemical attribute that must be carefully considered is the C/N ratio of organic matter (Table 1). When wastes are used in low fertility soils, N will be immobilized for an extended period due to incorporation into the microbial biomass or particularly when sludge has a higher C/N ratio, with potentially negative consequences for seedlings and initial vegetation. Fertilization with a readily available N source may alleviate the period of this immobilization or may speed up organic waste degradation by microbes in soil, because the C/N ratio will be reduced. On the other hand, slower degradation of the organic wastes may provide the best long term benefit as leaching losses would be minimized and N inputs more closely resemble that in natural soils, as was found with yard organic wastes that initially led to net immobilization (Quaggio, 2000). Therefore, the C/N ratio will guide the process of N mineralization or immobilization in the soil.

Chemical elements

The wastes had a high Ca concentration. However, the concentration was lower in RF (Table 2). The content of this element in RL, RM, RP, RC and RF was approximately 15.19, 2.20, 21.31, 11.45 and 1.52 times, respectively, and higher than the element Mg (Table 2).

This ratio is high when compared to the dolomitic limestones having an average of 28% Ca and 12% of Mg (Claassen and Carey, 2004), except in RM and RF. A high Ca/Mg ratio may lead to a lack of Mg in the plants, if the product is added in large quantities to soils with low availability of this element (Nelson et al., 1998). This is due to competition between Ca and Mg absorption by the sites present in the plasma membranes of plant roots. The high Ca content in some wastes is due to the addition of this element in calcium oxide form (CaO) to achieve better stabilization and reduction of pathogens and vectors.

The micronutrients and trace-elements (Table 2) in all wastes studied were lower than the maximum permissible concentration (MPC) in biological sludge recommended for agricultural use (CONAMA, 2006; USEPA, 1993; MAPA, 2006) (Table 3).

Conclusions

The pH in RM and RF were characterized as acid, showing no ability to ameliorate soil acidity; However, RL which was considered as neutral and RL and RC which were considered as alkaline increased soil pH, producing excellent results in correcting soil acidity. The DSS and FPW organic wastes would work well for vegetation that requires significant N inputs as it provided a steady and significant amount of N throughout the 112 days. In settings where available N could be detrimental, such as native plant restorations, or in other instances where weed pressure is undesirable, PS and USS application could be a simple way to decrease available N in the short term, but it would most likely provide a slowly available source over the longer term. The PMS organic waste could be an immediate carbon source and provide nitrogen after 70 days. Restoration of native perennial plant communities has been achieved through a high C/N ratio of organic soil amendments, such as sucrose and

sawdust that limit available N.

The C/N ratios of the wastes are different and as such, the RL and RF would work well for vegetation that requires significant N inputs as it provided a steady and significant amount of N throughout the 112 days. In settings where available N could be detrimental, such as native plant restorations, or in other instances where weed pressure is undesirable, RP and RM application could be a simple way to decrease available N in the short term, but it would most likely provide a slowly available source over the longer term. RC could be an immediate carbon source and provide nitrogen after 70 days. Restoration of native perennial plant communities has been achieved through a high C/N ratio of organic soil amendments, such as sucrose and sawdust that limit available N (Paschke et al., 2000).

All organic wastes were present in high concentrations of Ca, Mg, organic C, P, total-N and mineral-N. The levels of micronutrients (Cu, Fe and Mn) are satisfactory, without any risk of phytotoxicity and the heavy-metal concentration was low, indicating that initially they can be used as organic fertilizers.

ACKNOWLEDGEMENTS

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