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Communications in Soil Science and Plant Analysis

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/lcss20>

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Accepted author version posted online: 28 Jan 2015. Published online: 28 Jan 2015.

To cite this article: Arci D. Wastowski, Vanderlei R. da Silva, Maurício R. Cherubin, Moacir T. de Moraes, João P. G. Rigon, Fernando Arnuti, Genesio M. da Rosa & Paulo R. B. da Silva (2015): Chemical Characterization of Organic Residues in Agroindustrial and Urban Waste using Energy Dispersive X-ray Fluorescence Spectrometry, Communications in Soil Science and Plant Analysis, DOI: [10.1080/00103624.2015.1005217](https://doi.org/10.1080/00103624.2015.1005217)

To link to this article: <http://dx.doi.org/10.1080/00103624.2015.1005217>

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Chemical Characterization of Organic Residues in Agroindustrial and Urban Waste using Energy Dispersive X-ray Fluorescence Spectrometry

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Treated and reused industrial residues and urban waste are depicted as potential alternative sources for agricultural use. In this context, the objective of this study was to determine the chemical element concentrations in organic residues from the Medium-High Uruguay Region of the Rio Grande do Sul, using energy dispersive x-ray fluorescence spectrometry (EDXRF). The organic compounds analyzed were meat agroindustry sludge, organic composites from agribusiness meat, and organic compounds from urban waste (particle sizes: >4.75 mm, <4.75 mm to >2.00 mm, and <2.00 mm). The results showed that chemical elements found in greater concentrations were calcium, silicon, iron, aluminum, phosphorus, sulphur, and potassium. Thus, the EDXRF technique proves to be efficient for characterizing organic compounds and that these residues are appropriate as alternative sources of organic fertilizer that can be applied in an efficient and safe manner in agricultural systems.

Keywords Energy-dispersive x-ray fluorescence spectrometry, multielement analysis, organic fertilizer, organic wastes

Introduction

Due to human activity, large amounts of organic residue are generated daily. Therefore, adequate use in agricultural activities requires the characterization of the main properties of this organic residue. Chemical and physical characterization is important when planning the use and management of organic residue (Higashikawa, Silva, and Bettiol 2010). The growth of large urban centers has led to a disorderly increase in residue generation and consequently acceleration in the pace of natural resource degradation. The reduction of environmental impacts caused by urban garbage and industrial effluences is one of the major challenges to be faced by humankind in the twenty-first century (Abreu et al. 2005).

Received 20 March 2012; accepted 1 July 2014.

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In recent years, studies that focus on the correct disposal of these residues, so that besides ceasing to be an environmental problem they can become an economically feasible alternatives for application in agriculture, have increased. Organic compost from agroindustry residue can be used in agriculture production (annual and perennial crops); in reforestation areas, parks, and gardens; and to recover areas degraded by mining, landfills, and soil erosion (Matos 2005).

The organic fraction of urban waste can be treated through composting, with the final product resulting in wet organic residue with potential use in agriculture (Abreu et al. 2005). Thus, knowledge of the chemical makeup of these residues becomes indispensable. The composition of these residues varies as a result of the type of effluent treated, the treatment process used, and the quality of material generated (Bettiol and Camargo 2000). These residues should thus be evaluated on a case-by-case basis in terms of their agronomic value and the possible effects on soil quality so they can be applied in an efficient and safe manner in agriculture systems.

With the objective of strengthening the chemical analysis and characterization process of these residues using faster and easier analyses, the energy dispersive x-ray fluorescence spectrometry technique (EDXRF) stands out as an important alternative with growing use in the chemical characterization of several materials, such as soils (Dantas et al. 1981; Wastowski et al. 2010), rocks (Albers et al. 2002; Ferreira et al. 2003), ceramics (Bona, Sarkis, and Salvador 2007), biological materials (Adeloju, Bond, and Briggs 1985), air-suspended particles (Moraes 2009), and liquids (Pataca, Bortoleto, and Bueno 2005).

The conventional spectrometer (EDXRF) consists of just two basic units, the source of excitation and the spectrometric detection system (Figure 1). A high-resolution semiconductor detector is normally used (Si(Li)), because the resolution of the dispersive energy system is directly due to the detector's intrinsic resolution.

The principle for the technique's operation is based on the atomic ionization process and the emission of characteristic x-rays (Figure 2) where (a) the atom is submitted to an irradiation process by induction of a particle or by an x-ray source; (b) a photon with more energy than the connection energy ejects an electron from the atom's internal electronic layer; (c) one part of its energy can be absorbed by rearranging electrons (photoelectric absorption) and the atom is ionized; and (d) there is a rearrangement of atom electrons so the vacancy left can be filled by an electron from the most external electronic layers, releasing the existing difference in energy between the two levels of energy involved. That is how the characteristic emission of x-rays occurs (fluorescence). Because this process involves levels of energy that are characteristic of each element, the radiation emitted for

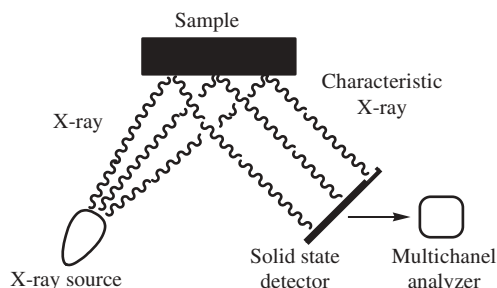


Figure 1. Basic schematic for an x-ray fluorescence spectrometer (EDXRF).

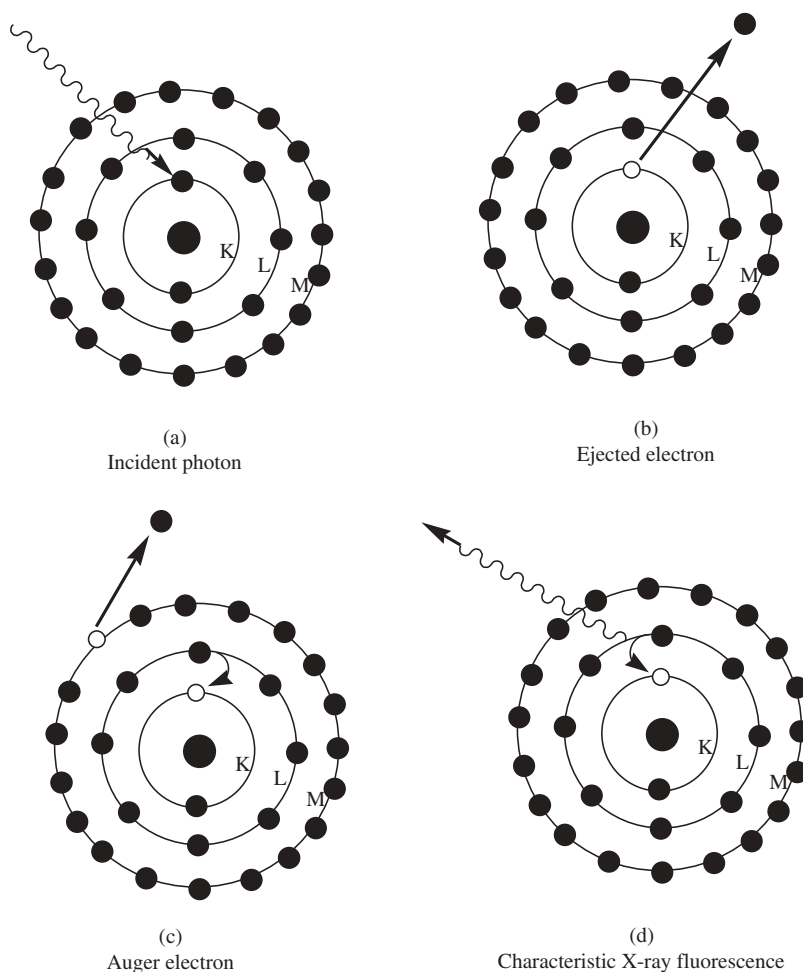


Figure 2. Representation of x-ray fluorescence essentials.

each transition is also characteristic. In this sense, emitted radiation energy can be directly employed to identify the element in question. Furthermore, because the intensity of emitted radiation is a result of element concentration, the technique also provides quantitative information (Shoog et al. 2009).

The technique thus permits simultaneous or sequential determination of the concentration of all elements between sodium (Na) and uranium (U), without destroying the sample, that is, in an instrumental manner, without any chemical pretreatment. It is able to reach detection limits of 1 to 20 ppm (Boman et al. 1996; Mamani 2003; Nagata, Bueno, and Peralta-Zamora 2001).

In this context, this study aimed at characterizing the levels of chemical elements in organic residue from the Medium-High Uruguay Region, RS, with potential for use in agriculture, using the energy dispersive x-ray fluorescence spectrometry technique (EDXRF).

Materials and Methods

The study was conducted at the Chemical Analysis and Research Laboratory (LAPAQ) of the Federal University of Santa Maria, Frederico Westphalen Campus, RS. The three organic residues analyzed are produced in the Medium-High Uruguay region, RS, at a meat agroindustry and the Intermunicipal Solid Waste Management Consortium (CIGRES): (a) meat agroindustry sludge, a residue composed of liquid swine dejects (manure), blood, solubilized fat, seasonings, and salt that underwent an anaerobic decomposition process with correction of pH up to 7.0 and oxygenation for transforming ammonia ion (NH_4^+) and ammonia (NH_3) into nitrate (NO_3^-) and nitrite (NO_2^-); (b) organic composites from meat agroindustry effluent, which is material obtained after composting and biochemical stabilization of the mixture, fraction in suspension (greases and oils) of the liquid in treatment, burnt wood residue (ashes), calcium hydroxide [$\text{Ca}(\text{OH})_2$; hydrated lime], and sawdust; and (c) organic residue from urban waste, which is material obtained from composting of the organic fraction of urban waste separated manually in a solid-waste recycling plant.

After collection, the samples were dried in a forced-air circulation kiln at a temperature of 105 °C for 48 h, calculating the moisture content. Later, the dried samples were ground manually in a porcelain crucible. Approximately 3 g of organic residue samples were used, packed on a 6- μm thick Mylar film, and stretched on the bottom of a 32-mm-outer-diameter and 23-mm-high polyethylene cell.

Analyses of the chemical elements were conducted using a Shimadzu EDX-720 energy dispersive x-ray fluorescence spectrometer. The following equipment operation conditions were chosen: tube voltage of 15 keV (Na to Sc) and 50 keV (Ti to U) with tube current at 184 μA and 25 μA respectively, a 10-mm collimator, 300-s real time integration, 40 percent and 39 percent dead time detector, under vacuum and Si(Li) detector refrigerated with liquid nitrogen. The analytical method used is called the fundamental parameters (FP) method. This method permits obtaining the equipment's sensitivity curve for each element of interest when a sample of a known chemical composition is submitted to well-defined instrumental parameters. The equipment's sensitivity curve relates calculated theoretical fluorescent intensity and the measure for each element (Bona et al. 2007).

In general, quantitative analysis by EDXRF is conducted using the calibration curve method, obtained with many standards. However, for some applications, it is difficult to obtain enough certified standards with matrixes similar to the samples and to achieve good distribution of data points on the scale of each element to be determined (Marguí et al. 2009; Omote, Kohno, and Toda 1995). The experimental analysis results for several different samples (Han et al. 2006) proved that high-precision results can be obtained using the FP method, even if only samples of pure elements were used for calibration. This also clearly illustrates that the FP method can effectively correct the complicated calculation of the matrix effect. Therefore, for a mass routine analysis, if the calibration samples are absent, the FP method can provide relatively precise and quantitative results.

Element sensitivity is generally determined by measuring the characteristics of x-rays emitted by standards that only have one element. The use of commercially available standards prepared in a laboratory using pure or composite elements has proven to be efficient for determining element sensitivity in x-ray fluorescence systems (XRF), because they are cheap and can be easily prepared (Lopes 1989). To correct absorption effects and calibrate the spectral lines of analyzed elements, standard A-750 was used. The calibration standard used, provided with the EDX-720 equipment, is a metal alloy composed of aluminum, tin, magnesium, iron, and copper. It is thus possible to determine the composition of the material analyzed.

Results permit determining the concentration of existing sodium (Na) and uranium (U) chemical elements in analyzed organic residue, as well as the fraction of their organic and mineral material.

Statistical methods were applied to analyze and point out values of the elements in studied. The mean was used for statistical analysis and to determine the degree of different metal concentrations.

Results and Discussion

Table 1 shows the results for dry matter, moisture content, and organic and mineral material of the three analyzed residues. It was verified that meat agroindustry sludge moisture content was 90 percent. This result is similar to the values obtained by Trannin, Siqueira, and Moreira (2005), who in 2 years of studies with industrial sludge observed moisture contents of 80 and 82 percent. However, moisture content in meat agroindustry organic composites was 57.87 percent, a greater percentage of dry mass in constitution (42.13 percent) when compared to sludge, a fact that is explained as a result of the mixture of effluent residue with materials with less moisture (ashes, hydrated lime, and sawdust).

Moisture contents obtained in urban waste residue (granulometry brackets >4.75 mm, 4.75 mm to 2.00 mm, and <2.00 mm) were 44.29 percent, 49.95 percent, and 54.61 percent, respectively. In a classification of chemical characteristics for urban waste residue, moisture contents greater than 35 percent are considered excessive (Abreu et al. 2005). This high moisture characteristic ends up hindering mechanized management for applying this residue in crops.

As to the percentages of organic matter and mineral matter, the organic composite of the meat agroindustry effluent was seen to have the lowest and highest contents (67.90 percent and 32.10 percent), respectively. These results are justified as a result of adding material composed of burnt wood residue (ashes) and hydrated lime, which contain high fractions of mineral matter. In urban waste residue, an increase in mineral matter was seen as the granulometry diminished, a fact that is probably tied to the material composting process. Thus, at the biggest fraction (diameter > 4.75 mm), thicker organic matter is present that does not suffer complete mineralization in composting, as is plastic matter, which could be mixed in with the residue because the waste does not undergo a selective collection process but only a manual gathering on a conveyor belt.

Table 1

Total dry matter (MS), moisture (U), organic material (MO) and mineral materials (MM) of meat agroindustry sludge (LAC), organic composite of meat agroindustry effluents (COEAC), and organic residue of urban waste (ROLU) (Frederico Westphalen, RS, Brazil, 2010)

Residue	MS (%)	U (%)	MO (%)	MM (%)
LAC	10.00	90.00	87.82	12.18
COEAC	42.13	57.87	67.90	32.10
ROLU (>4.75 mm)	55.71	44.29	90.08	9.92
ROLU (4.75 mm to 2.00 mm)	50.05	49.95	87.82	12.18
ROLU (<2.00 mm)	45.39	54.61	82.52	17.48

In the chemical characterization of organic residue, conducted using the EDXRF technique, a total of twenty-three chemical elements were detected, including sodium (Na) and uranium (U), as shown in Figure 3.

Table 2 shows the results for the chemical element concentrations detected in the residue, separating them into three groups: macronutrients, heavy metals, and other elements. In organic residue of urban waste, on average in the different granulometric fractions, the macronutrient calcium stood out with the greatest concentrations, ranging from 21.85 to 43.72 g kg⁻¹. Then, the following elements were found in greater concentrations: sulfur, phosphorus, and potassium. Sulfur was the only element that presented a reduction in concentration as a result of the reduction in granulometry of the material, ranging from 6.13 g kg⁻¹ (sieve > 4.75 mm) to 5.42 g kg⁻¹ (sieve < 2 mm). Phosphorous and potassium accumulated as a result of a reduction in diameter of organic residue of urban waste, but they did not present variation in concentration between the two macronutrients. For phosphorus, the concentration ranged between 3.22 g kg⁻¹ (sieve > 4.75 mm) and 6.42 g kg⁻¹ (sieve < 2 mm), and potassium presented values of 3.90 g kg⁻¹ (sieve > 4.75 mm) to 5.15 g kg⁻¹ (sieve < 2 mm). It was thus possible to observe the nutrition potential this organic residue of urban waste has for use in agriculture.

With regard to meat agroindustry sludge, there was a concentration similar to calcium (19.92 g kg⁻¹), sulfur (18.87 g kg⁻¹), phosphorus (14.35 g kg⁻¹), and potassium (16.60 g kg⁻¹) elements; thus, we can see that this concentration is appropriate for fertilizing agricultural crops.

From the results obtained in the characterization of organic residue from meat agroindustry effluents, the concentration of calcium (228.56 g kg⁻¹) was observed as being much greater than the concentration of other elements. However, this high concentration can be attributed to the addition of hydrated lime in composite formulation. Besides the high concentrations of calcium that could direct the use of this composite to agriculture, as a source of Ca²⁺, it is also necessary to underscore phosphorus and potassium contents (39.95 and 15.01 g kg⁻¹ respectively), which are fundamental elements for plant development.

Elements classified as heavy metals were found in contents less than pollutant limits. Of the fourteen elements considered heavy metals, iron, manganese, titanium, and zinc were observed in all characterized materials, and arsenic, cobalt, mercury, molybdenum, and selenium was not observed in any of the analyzed residues. The other heavy metals were detected in at least one of the residues studied.

When analyzing the materials separately, heavy-metal content is checked in organic residue of urban waste from meat agroindustry sludge. Polluting levels are not observed, demonstrating that the treatment system is efficient and guarantees effluent production with proper agronomic characteristics for use in farms.

As to the limits of heavy metals tolerated in urban waste residue, Brazil does not have legislation in effect for regulating it. However, the levels found in the different granulometries of the urban waste residue are less than the critical levels established by several countries such as the United States, Spain, Holland, Italy, and Switzerland (Abreu et al. 2005), thus demonstrating agronomically appropriate characteristics for use as an alternative source of agriculture fertilizer.

However, even at nonpolluting levels, a tendency can be observed for an increase in concentrations of existing heavy metals as particle size was reduced. These results can be attributed to greater mineralization of these elements in the material composting process, as well as the presence of polluting materials improperly mixed in with the residue. In Brazil, the factor that leads to the production of low-quality organic waste residue is mainly related to the presence of organic and inorganic pollutants, where the improvement of this product

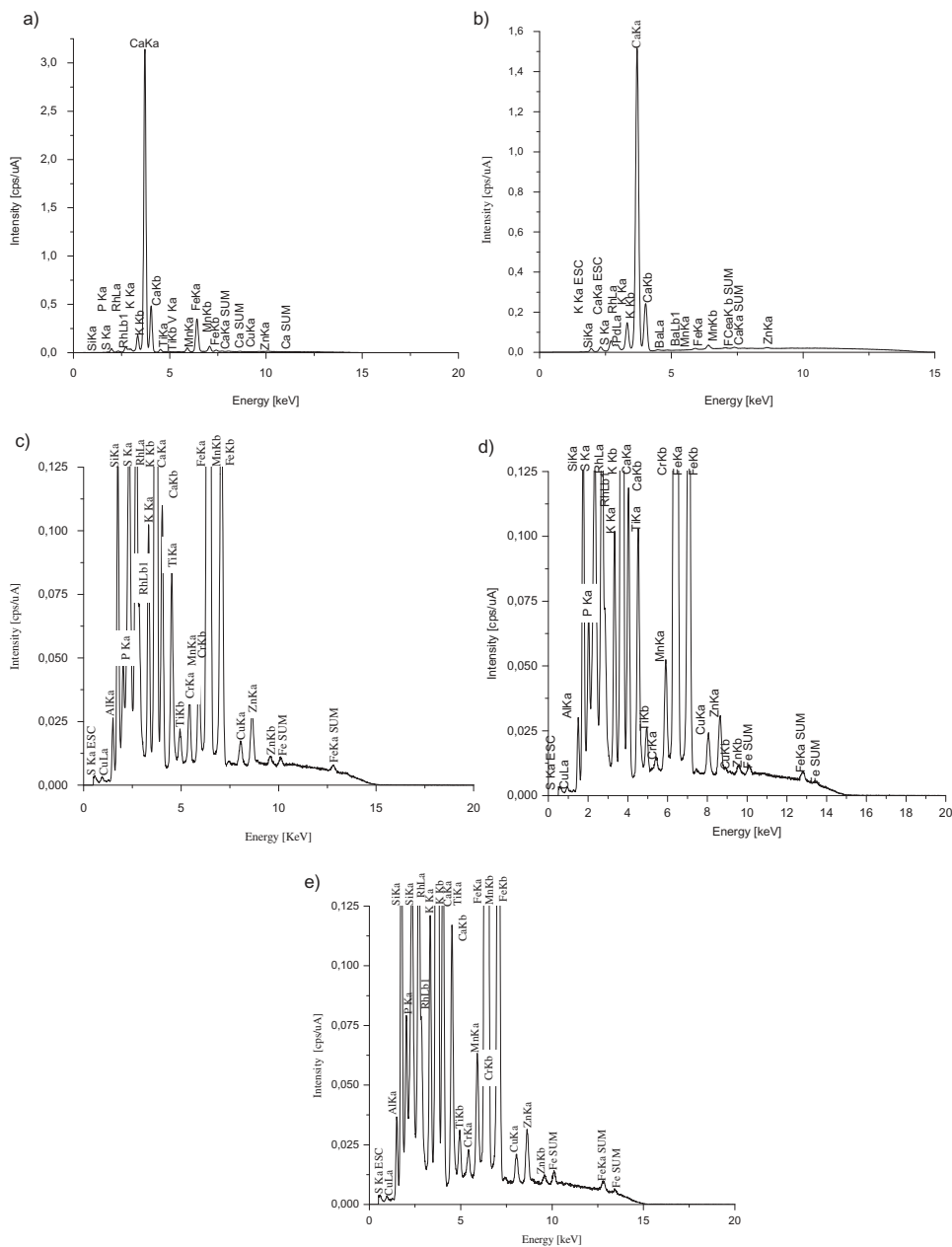


Figure 3. Characteristic x-ray spectrum for the samples (a) for meat agroindustry sludge; (b) for organic composites of the meat agroindustry effluent; (c) for organic residue of urban waste with granulometry greater than 4.75 mm; (d) for organic residue of urban waste with granulometry of 4.75 to 2.00 mm; and (e) for organic residue of urban waste with granulometry under 2.00 mm, detected using the EDXRF technique (Frederico Westphalen, RS, Brazil, 2010).

Table 2

Analysis of the chemical composition of meat agroindustry sludge, organic composite of meat agroindustry effluents, and organic residue from the urban waste recycling plant (Frederico Westphalen, RS, Brazil, 2010)

Material	Different granulometry of organic residue of urban waste (mm)			Meat agroindustry sludge	Organic composite of meat agroindustry effluent
	>4.75	475 to 2	<2		
Macronutrients					
Calcium (g kg ⁻¹)	21.85	24.78	43.72	19.92	228.56
Sulfur (g kg ⁻¹)	6.13	4.77	5.42	18.87	3.51
Phosphorus (g kg ⁻¹)	3.22	5.23	6.42	14.35	39.95
Potassium (g kg ⁻¹)	3.90	4.03	5.15	16.60	15.01
Heavy metals					
Arsenic (g kg ⁻¹)	nd	nd	nd	nd	nd
Cadmium (g kg ⁻¹)	nd	nd	0.035	nd	nd
Lead (g kg ⁻¹)	nd	nd	0.029	nd	nd
Cobalt (g kg ⁻¹)	nd	nd	nd	nd	nd
Copper (g kg ⁻¹)	0.180	0.252	0.319	1.72	nd
Chrome (g kg ⁻¹)	0.421	0.087	0.235	0.060	nd
Iron (g kg ⁻¹)	16.85	26.94	38.120	19.66	13.14
Manganese (g kg ⁻¹)	0.515	0.760	1.050	0.780	1.62
Mercury (g kg ⁻¹)	nd	nd	nd	nd	nd
Molybdenum (g kg ⁻¹)	nd	nd	nd	nd	nd
Nickel (g kg ⁻¹)	0.013	nd	nd	nd	nd
Selenium (g kg ⁻¹)	nd	nd	nd	nd	nd
Titanium (g kg ⁻¹)	1.58	2.59	3.41	1.36	1.71
Zinc (g kg ⁻¹)	0.412	0.503	0.665	3.06	0.161
Other elements					
Aluminum (g kg ⁻¹)	17.22	19.87	25.84	14.47	nd
Actinium (g kg ⁻¹)	0.025	nd	0.022	nd	nd
Strontium (g kg ⁻¹)	0.076	0.080	0.187	0.17	1.454
Barium (g kg ⁻¹)	0.804	0.80	0.982	0.320	1.15
Yttrium (g kg ⁻¹)	nd	0.013	0.017	nd	nd
Silver (g kg ⁻¹)	nd	nd	0.077	nd	nd
Rubidium (g kg ⁻¹)	nd	nd	nd	nd	0.042
Silica (g kg ⁻¹)	26.01	31.07	42.54	24.77	14.66
Thulium (g kg ⁻¹)	nd	nd	0.569	nd	nd
Zirconium (g kg ⁻¹)	0.034	0.067	nd	nd	nd

Note. nd, not detected (concentration under 1 mg kg⁻¹).

would occur through the selective collection of household garbage, where obtaining the material does not place the environment at risk through contaminations (Egreja Filho et al. 1999; Grossi 1993).

Besides the aforementioned elements, using the EDXRF technique for analysis, it was possible to detect low levels of nine more elements in some samples, which are manganese, barium, thulium, strontium, silver, yttrium, zirconium, and rubidium, thus demonstrating it is an efficient multielement analysis technique to determine and quantify levels of chemical elements present in organic residue.

Conclusions

Energy-dispersive x-ray fluorescence spectrometry (EDXRF) can be used to characterize levels of chemical elements present in the organic residue of urban waste, sludge, and organic composites of meat agroindustry effluents.

The chemical elements found in greater concentrations in analyzed organic residues were calcium, silica, iron, aluminum, phosphorus, sulphur, and potassium, demonstrating that the residues evaluated can be considered alternative sources of organic fertilizers and can be applied efficiently and safely in agriculture systems.

Analyzed residues do not have polluting levels of heavy-metal contents, giving them agronomically appropriate characteristics for use in agriculture.

Acknowledgments

The authors acknowledge the study materials provided by the following companies: Frigorífico Mabella Carnes and Marfrig Alimentos S/A and the Intermunicipal Solid Waste Management Consortium (CIGRES).

Funding

The authors thank the National Council on Scientific and Technological Development (CNPq).

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