REVISTA BRASILEIRA DE ENERGIAS RENOVÁVEIS

EVALUATION OF THE ELEMENTAL COMPOSITION OF MUNICIPAL SOLID WASTE BOTTOM ASH: A NEW METHODOLOGY FOR SAMPLE PREPARATION[1]

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[1] Apresentado no Congresso de Energias Renováveis da UFSCar: 23 a 26 de maio de 2017 – Sorocaba-SP, Brasil

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ABSTRACT

The disposal of municipal solid waste (MSW) to thermochemical treatments promotes the formation of ashes as a by-product, which constitutes an important role in the design and operation of energy recovery plants, as the ash can cause corrosion and fouling problems. In this sense, this work analyzed samples of bottom ashes from combustible fractions (organic matter, plastics, textiles, paper/cardboard/Tetra Pak® and sanitary waste) of MSW *in natura* from Santo André – SP, Brazil. For this, a new methodology for sample preparation was proposed to evaluate the elemental composition of the bottom ashes, for later analysis by Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDS). The obtained data showed a standard deviation of less than 10%, guaranteeing a greater reliability

of the results and corroborating with standard deviations presented in the scientific literature. In addition, the elemental composition of the bottom ash was similar between the different combustible fractions evaluated.

Keywords: MSW, Combustible fractions, SEM-EDS.

AVALIAÇÃO DA COMPOSIÇÃO ELEMENTAR DE CINZAS DE RESÍDUOS SÓLIDOS MUNICIPAIS: UMA NOVA METODOLOGIA PARA PREPARAÇÃO DE AMOSTRA

RESUMO

A eliminação de resíduos sólidos urbanos (MSW) para tratamentos termoquímicos promove a formação de cinzas como subproduto, o que constitui um papel importante no projeto e operação de plantas de recuperação de energia, já que a cinza pode causar problemas de corrosão e contaminação. Nesse sentido, este trabalho analisou amostras de cinzas de frações combustíveis (matéria orgânica, plásticos, têxteis, papel / cartão / Tetra Pak® e resíduos sanitários) de MSW in natura de Santo André - SP, Brasil. Para isso, foi proposta uma nova metodologia para a preparação da amostra para avaliar a composição elementar das cinzas, para análise posterior por microscopia eletrônica de varredura com espectroscopia de raios-X com dispersão energética (SEM-EDS). Os dados obtidos mostraram um desvio padrão inferior a 10%, garantindo uma maior confiabilidade dos resultados e corroborando os desvios padrão apresentados na literatura científica. Além disso, a composição elementar da cinza foi semelhante entre as diferentes frações combustíveis avaliadas.

Palavras-chave: MSW, Frações Combustíveis, SEM-EDS.

INTRODUCTION

Municipal solid waste (MSW) are products derived from anthropogenic residential, commercial and institutional activities. It includes, among others, organic matter, plastic packaging, papers, textiles, metals and glass, which must be managed by municipalities. Its gravimetric composition varies according to socioeconomic and cultural conditions, local laws and seasonality (BAAWAIN et al., 2017; EPA, 2017; HONG et al., 2017).

The practice of management related to MSW emerged from the need to control public health problems related to the proliferation of diseases in urban centers, as well as reduce the volume of accumulated waste. With the rapid growth of the world population and the increase in the *per capita* generation of waste, the main objective of the management evidenced the appropriate handling of MSW (BRUNNER; RECHBERGER, 2015; CUCCHIELLA et al., 2014; SAIANI et al., 2014).

It is estimated that the global MSW generation is 1.3 billion tons per year (HONG *et al*., 2017). In 2015, for example, ~242.7 million tons of MSW were generated in Europe (< 0.1% higher than in 2013) and in Brazil this amount was ~79.9 million (15.8% higher than in 2013) (ABRELPE, 2013; ABRELPE, 2015; ARACIL et al., 2017; EUROSTAT, 2017).

Many environmental and social problems are related to inadequate disposal of MSW, such as, for example, contamination of soil and groundwater with chemical and organic compounds (leached), harmful to the environment, due to the existence of open dumping. The intense economic activities and the lack of qualified training in modern MSW management intensify these problems (BAAWAIN et al., 2017; KORAI et al., 2017).

Moreover, even with the technologies available for treatment and disposal (landfill, mechanical-biological treatment, among others), not all the amount of waste generated can have its final dispose in an appropriate place. In this way, a possible alternative to a destination of waste could be to thermochemical treatments (direct combustion, gasification and pyrolysis), aiming reduction of waste volume with concomitant energy recovery (GUNASEE et al., 2016; LONARDO et al., 2016).

In direct combustion processes in energy recovery plants, bottom ash (slag) and volatile ash (retained in filter systems) are generated as by-products (VALORSUL, 2017). In incineration, for example, the highest percentage of ash corresponds to bottom ash (15-35% by mass of treated waste) (ALLEGRINI et al., 2014; UNEP, 2017). Although the percentage of the mineral fraction (inorganic material) is lower than the carbonaceous matrix of a fuel, it constitutes an important role in the design and operation of energy recovery plants, as the ash can cause corrosion and fouling problems, as well as negatively influence the fuel potential (GRAMMELIS, 2011).

In this context, the characterization of ashes is of paramount importance, especially in relation to its composition. There are analytical techniques that allow this evaluation, such as: X-ray Fluorescence-XRF; Inductively Coupled Plasma-ICP-MS; X-ray diffraction-XRD;

and Scanning Electron Microscopy with Dispersive X-ray Spectroscopy (SEM-EDS). This last technique was used in this work, with the objective of obtaining information on the composition of the *in natura* MSW ash from the municipality of Santo André – SP, Brazil.

MATERIALS AND METHODS

Obtainment of samples

The samples of *in natura* MSW were from the municipality of Santo André – SP and were obtained from the proximate analysis for fractions of organic matter, plastics, textiles, paper/cardboard/Tetra Pak® and sanitary waste.

Preparation of the samples

As samples of the different MSW fractions were quartered, homogenized, milled in landfill of Santo André (particle size standardization to 0.8 cm) and dried until equilibrium moisture content, according to Gutierrez (2016). After the proximate analysis of the different fractions, the resultant ash were stored in polypropylene Falcon® tubes of 15 mL.

Previously, the ashes were processed inside the storage tubes, using a pre-sanitized glass rod with acetone (HPLC grade), in order to reduce particles size. The stubs were also properly sanitized with acetone to avoid contamination of the samples with impurities, as well as to ensure total evaporation of the substance, avoiding problems with the fixation of carbon tape. These were demarcated in three sections for the analyses, as three replicas for each evaluated combustible fraction.

The roughly pulverized samples were deposited, with the assistance of a spatula, over double-coated carbon tapes (~7 x 8 mm), previously fixed to the marked sections (with the aid of tweezers) in different types of stubs: i. aluminum stubs for samples of organic matter, plastics, textiles and sanitary waste; ii. carbon stub for paper/cardboard/Tetra Pak®. It is emphasized that the quantification of aluminum present in this fraction could be compromised when using an aluminum stub.

To ensure a uniform deposition of material for subsequent gold coating, avoiding the contamination of the microscope detectors by the dispersion of low-density particles inside

the equipment, the excess sample was removed. It could be noted that this procedure was performed for each combustible fraction.

The prepared stubs were stored in a glass desiccator for further sputter coating with gold (15 nm in diffuse mode; *Leica EM ACE200*). It should be noted that the coating is of great importance to ensure the excess of electrons flow in the samples (analysis by SEM-EDS), but it must also have adequate thickness, as to enable the interaction between the electron beam and the sample. Figure 1 illustrates the steps involved in preparing the samples for evaluation of the elemental composition of the bottom ash of MSW.

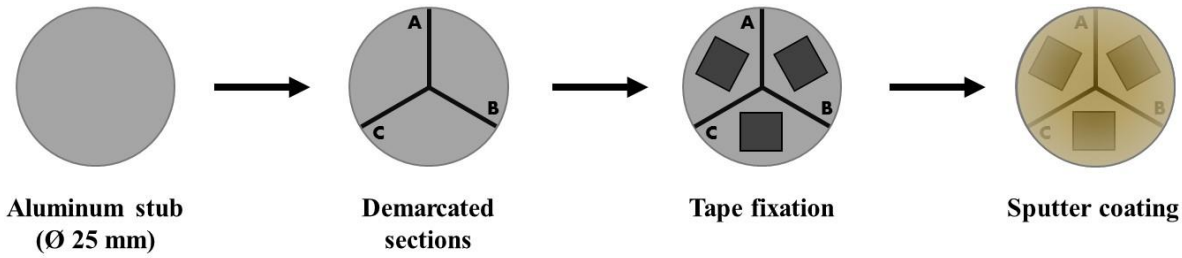

Figure 1: Subsequent steps for sample preparation.

Analysis of elemental ash composition of MSW

The analyses were performed using the SEM-EDS technique (*JCM-6000-OPT Neoscope II EDS Analysis Option*). The operating conditions of the equipment were: high vacuum, secondary electron detector; 15 kV voltage; high filament intensity; and high current density. Spectra were obtained from an image standardization with magnification of 200 times. For each stub, three points were randomly selected by section. That is, the test was performed as nine replicas for each evaluated fraction.

RESULTS AND DISCUSSION

The proposed methodology for the preparation of ash samples of MSW for further analysis by SEM-EDS aims to obtain accurate results on the elemental composition of the bottom ash of combustible fractions, with low standard deviation (<10%), ensuring greater result reliability. According to Jansen et al. (2004), data of characterization for samples of MSW shows a standard deviation between 3 and 10%. And, according to Table 1, it is

possible to observe that the data on the elemental composition of the ashes of the plastic fraction have standard deviations of less than 10%, except for chlorine (~19%). It is suggested that the greatest deviation found is due to the fact that this element is present at a low concentration, with detection level near the lower limit of the equipment which, according to the manufacturer, has 1% error.

Table 1: Elemental composition of replicas of plastic bottom ash from MSW.

Elements (% by mass)	A			B			C			Mean	SD
	001	002	003	001	002	003	001	002	003		
C	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	
Na	5.94	6.58	6.28	7.26	7.09	5.94	6.33	6.30	6.43	6.46	0.43
Mg	7.99	8.40	8.24	9.26	8.42	8.48	7.97	7.54	8.50	8.31	0.45
Al	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	
Si	18.89	18.97	18.75	19.71	19.44	20.96	19.07	19.29	18.96	19.34	0.64
P	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	
S	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	
Cl	1.03	1.20	0.99	1.13	0.99	1.74	1.07	1.13	0.99	1.14	0.22
K	2.86	2.44	2.48	2.61	2.56	2.70	2.55	2.67	2.31	2.57	0.15
Ca	35.09	34.09	34.66	33.22	33.62	36.06	36.41	35.12	34.70	34.77	0.99
Ti	18.39	19.05	19.44	17.18	18.76	16.43	18.96	18.59	19.54	18.48	0.98
V	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	
Fe	8.62	8.50	8.12	7.50	7.39	7.17	7.45	8.26	7.37	7.82	0.52
Zn	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	
Au	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	

nd: not determined; ni: not identified.

Among the advantages that can be highlighted due to the use of the SEM-EDS technique, stands out that the MSW samples do not need to be digested, besides the no need of the use of commercial standards and construction of calibration curves, as it happens for analysis of ICP-MS (MAGDZIARZ et al., 2016), adding savings of financial resources, time of analysis, labor, among other factors.

Table 2 shows the mass percentage of the elements determined in the bottom ash (metals, semimetals and halogens) of the MSW combustible fractions evaluated, with the most abundant ones being: organic matter (Ca, Si, Mg, Cl, K, Na, Fe, P), plastics (Ca, Si, Ti, Mg, Fe), textiles (Ca, Si, Mg, Fe, Na), paper/cardboard/Tetra Pak® (Ca, Al, Si, Mg, Na) e sanitary waste (Na, Ca, Cl, Ti, Si).

Table 2: Elements determined in bottom ash of MSW combustible fractions.

Elements (% by mass)	Organic matter		Plastics		Textiles		Paper/cardboard/ Tetra Pak®		Sanitary waste	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
C	nd		nd		nd		nd		nd	
Na	6.53	0.51	6.46	0.43	7.12	0.28	3.36	0.38	33.07	1.63
Mg	9.41	0.62	8.31	0.45	8.38	0.50	3.42	0.20	2.45	0.20
Al	nd		nd		nd		14.60	3.44	nd	
Si	16.40	1.01	19.34	0.64	15.92	0.48	11.23	0.74	10.38	0.66
P	6.00	0.54	ni		ni		ni		ni	
S	ni		ni		4.40	0.28	ni		ni	
Cl	6.81	0.53	1.14	0.22	2.26	0.16	ni		13.95	0.99
K	6.67	0.37	2.57	0.15	5.29	0.25	2.26	0.28	8.20	0.47
Ca	39.96	0.89	34.77	0.99	44.04	1.39	61.93	2.90	17.65	0.76
Ti	ni		18.48	0.98	3.44	0.51	ni		11.60	1.30
V	ni		ni		ni		ni		ni	
Fe	6.22	0.22	7.82	0.52	7.92	0.49	1.45	0.40	1.56	0.40
Zn	ni		ni		ni		ni		ni	
Au	nd		nd		nd		nd		nd	

nd: not determined; ni: not identified.

In function of the stub type (aluminum or carbon), carbon tape and gold from the coating, the elements carbon gold and aluminum were disregarded (Table 2) of the organic matter, plastics, textiles and sanitary waste analyses. In the case of the paper/cardboard/Tetra Pak® samples, only carbon and gold were disregarded, as this fraction has an abundance of aluminum.

Among the metals, semimetals and halogens evaluated, the Ca is the element of the highest percentage for the fractions evaluated, except for sanitary waste, which the most abundant element was Na (Table 2).

The ashes, although thermally stable in some situations, undergo volatilization and condensation processes during and after combustion, which depend on the characteristics of the combustible (content and composition of the ash present, fixed carbon, volatile materials, density and particle size, etc.). It is noteworthy that the Na, K, P, Cl, Si and Ca contents determine the ash melting behavior – related to the formation of slag and fouling. However, analyzes of ash chemical composition do not indicate its behavior in terms of reactivity in combustion chambers (GRAMMELIS, 2011).

The importance of the quantification of S, Cl and K elements is related to the corrosion of equipment commonly used in energy recovery plants, such as heat exchangers. In combustion processes, S, Cl and K partially vaporize in high temperatures and, after cooling, form HCl, KCl, Cl_2, alkali chlorides and metal sulfides. In addition, some compounds may form deposits on the surfaces of pipe, reducing the heat transfer rate and efficiency of the energy conversion system. It should be noted that each 0.1% by mass (dry base) of chlorine in fuels, 100 ppm (by volume) of chlorine are formed in the gas phase (GRAMMELIS, 2011; MA, 2010 apud GALLEGO et al., 2014; MAGDZIARZ et al., 2016; SPLIETHOFF, 2010).

Yang et al. (2014) point out that the percentage of Cl has a significant variation in bottom ash from incinerated waste, corresponding to 0.2-5%, which corroborates with the values determined in this work, except for the fraction of sanitary waste (Table 2). Table 3 shows the oxides formed from the elements present in the evaluated combustible fractions.

Table 3: Oxides determined in bottom ash of MSW combustible fractions.

Compound(% by mass)	Organic Matter		Plastics		Textiles		Paper/cardboard/ Tetra Pak®		Sanitary waste	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Na_2O	6.52	0.37	5.47	0.35	6.13	0.23	2.92	0.33	31.14	1.61
MgO	7.36	0.38	8.64	0.45	8.87	0.52	3.66	0.23	2.85	0.25
Al_2O_3	nd		nd		nd		17.73	3.97	nd	
SiO_2	23.61	1.04	25.95	0.83	21.75	0.59	15.48	1.05	15.52	0.96
P_2O_5	7.58	0.94	ni		ni		ni		ni	
SO_3	ni		ni		7.02	0.43	ni		ni	
Cl	4.85	0.34	0.72	0.14	1.45	0.10	ni		9.46	1.11
K_2O	8.84	0.29	1.94	0.12	4.06	0.18	1.75	0.22	7.99	3.17
CaO	32.25	0.66	30.52	0.92	39.34	1.34	55.84	3.10	17.18	0.79
TiO_2	ni		19.35	1.03	3.66	0.55	ni		11.72	4.30
FeO	7.57	0.57	6.31	0.43	6.51	0.41	1.20	0.32	1.40	0.36
ZnO	ni		ni		ni		ni		ni	

nd: not determined; ni: not identified.

Reports were found (MAGDZIARZ et al., 2016; VASSILEV et al., 2010) which present the ash composition in different fuels, among which stand out the refuse-derived fuel (RDF) and biomass (Table 4).

Table 4: Mass percentage of compounds found in the literature in different ash samples.

Material	Compound (% by mass)										
	SiO_2	CaO	K_2O	P_2O_5	Al_2O_3	MgO	Fe_2O_3	SO_3	Na_2O	TiO_2	Cl

RDF	38.67	26.81	0.23	0.77	14.54	6.45	6.26	3.01	1.36	1.9	---
Biomass	21.50	40.0	9.85	3.77	1.00	4.66	1.19	1.4	0.04	0.07	0.08

Adapted from Vassilev et al., 2010; Magdziarz et al., 2016.

Comparing the biomass ashes and their most abundant compounds (Table 4), it is possible to observe that SiO_2 contents are close to those found in organic matter, plastics and textiles (Table 3). However, CaO has a similar percentage to the textile fraction, and K_2O is similarly found in organic matter and sanitary waste fractions (Table 3).

When comparing the RDF with the evaluated combustible fractions, it is assumed that Al_2O_3 composes a percentage similar to the paper/cardboard/Tetra Pak® fraction, and this similarity is given because Tetra Pak® packages have a high aluminum content in their composition, and this fraction is the most abundant in the RDF gravimetric composition (TIBURCIO et al., 2016).

Since RDF and several different biomasses are used in energy recovery systems by direct combustion, the values found for the solid waste analyzed in this work are close to those found in the literature, denoting similar characteristics and reliability in the obtained results. However, it is noteworthy that the ashes evaluated in this work did not undergo acid digestion, necessary to fix the chemical composition of the volatile ashes, and only the bottom ash was evaluated. The maintenance and complete evaluation of the solid waste used in energy recovery systems is indispensable in the evaluation of its use in an efficient way.

The scientific literature reports a growing concern about the recovery and valuation of ferrous metals and non-ferrous metals present in mineral form and aggregated to the ashes or in the form of scrap, and their potential uses in civil construction (paving of roads, concrete blocks, etc.) (ALLEGRINI et al., 2014; GARCIA-LODEIRO et al., 2016; LYNN et al., 2016; SAIKIA et al., 2015; SONG et al., 2015; TANG et al., 2015).

Due to the presence of different types of products found in MSW, the quantitative and qualitative evaluation of the elements that compose its ashes are of fundamental importance, since they have constituents that must be considered in the projection, construction and maintenance of equipment involved in combustion processes (for example, boilers and heat exchangers), increasing the life of such equipment. The compound elements found in diverse biomass ashes includes, among others, SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO,

Na_2O, K_2O, TiO_2, SO_3, P_2O_5, Cl, Br e Hg (KOPPEJAN, 2008; MAGDZIARZ et al., 2016; NIESSEN, 2002; VAN LOO).

CONCLUSIONS

In this work, ashes samples of organic matter, plastics, textiles, paper/cardboard/Tetra Pak® and sanitary waste from *in natura* MSW were analyzed using the SEM-EDS technique. Their elemental composition was determined, showing a standard deviation of less than 10%, guaranteeing a greater reliability of the results, besides corroborating with standard deviations presented in the literature (3-10%). However, a higher deviation was found for Cl in plastics fraction (~19%), indicating that the concentration of this element in the analyzed waste is below the detection level of the equipment.

From the information of the elemental composition of the ashes, it is possible to suggest corrosion and or corrosion indicators, depending on the characteristics of the waste in question and, consequently, to propose adaptations or new technologies to improve energy recovery systems.

In addition, it was found that the elemental composition of the bottom ash was similar between different combustible fractions, with Ca and Si as major elements, which are associated with the formation of slag and fouling in pipes. In the case of the sanitary waste fraction, by the presence of Na, it is possible that corrosion of the equipment involved in energy recovery systems occurs, due to the possible formations of $NaAlSiO_4$, NaCl e Na_2SO_4.

However, the ashes evaluated in this work did not undergo a process of acid digestion, necessary to chemically fix the volatile ashes, being only evaluated as bottom ashes. In this sense, a complete characterization of the ashes is necessary to evaluate their behavior, when volatilizable metals are emitted in systems of heat treatment.

ACKNOWLEDGEMENTS

This research was supported by Fundação de Amparo à Pesquisa do Estado de São Paulo – FAPESP (Process number 2011/51902-9), Agência Nacional de Energia Elétrica – ANEEL (Process number PD-0553-0022/2012), Petróleo Brasileiro S.A. – PETROBRAS and

Universidade Federal do ABC – UFABC (Term of Scientific and Technical Cooperation number 0050.0084633.13.9).

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