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Chemical parameters of gourmet salts compared withsalt extracted from *eichhornia crassipes* evaluated by energy-dispersive x-ray spectrometry

Estudo dos parâmetros químicos de sais gourmets quando comparados com sal extraído da *Eichhornia crassipes* avaliados por espectrometria de raios-X por dispersão em energia

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ABSTRACT

Salts have been part of human nutrition since 2,000 b.C., having originated in the Zhongba region, in China. Itssubstantial availability in food markets and stores specialized in condiments led us, curiously, to compare its elemental chemical composition with that of salt extracted from a floating aquatic macrophyte, known commonly as "water hyacinth" (*Eichhornia crassipes*), whose product, after extraction from dry matter, was popularly denominated"indigenous salt". The objective of the present study was to evaluate the chemical composition of gourmet salts in comparison with indigenous salt using the energy-dispersive X-ray spectrometry (EDX) technique. *Eichhornia crassipes* exhibited higher concentrations of chloride (35.834%), potassium (62.146%), sulfur (1.095%), calcium (0.450%), bromine (0.177%), silicon (0.124%), copper (0.029%), magnesium (0.123%), and rubidium (0.023%) than the gourmet salts. Our results indicate that this indigenous salt should be consumed under medical guidance due to its high concentration of potassium chloride, a salt whose properties are used for specific human treatment and not daily consumption.
Keywords: Gourmet salts; *Eichhornia crassipes*; Chemical Composition; EDX

RESUMO

Os sais fazem parte da alimentação humana desde os anos 2.000 a.C., originando-se inicialmente na região de Zhongba, na China. Em função da quantidade e disposição em mercados alimentícios e lojas especializadas em condimentos, levou-se assim, curiosamente, ao estudo de sua composição química elementar em comparação com um sal extraído de uma macrófita aquática flutuante conhecida como "aguapé" (*Eichhornia crassipes*), cujo produto após extração a partir da matéria seca denominou-se popularmente de "Sal indígena". O objetivo do presenteestudo foi de avaliar a composição química dos sais gourmets em comparação com este sal indígena utilizando a técnica de espectrometria de raios-X por dispersão em energia (EDX). A *Eichhornia crassipes* apresentou maiores conteúdos de cloreto (35,834%), potássio (62,146%), enxofre (1,095%), cálcio (0,450%), bromo (0,177%) silício (0,124%), cobre (0,029%), magnésio (0,123%) e rubídio (0,023%) quando comparada ao sal gourmet. Os resultados

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obtidos indicam queo sal indígena deve ser consumido sob orientação médica em função de sua elevada concentração de cloreto de potássio, um sal com propriedades específicas para tratamento humano e não para consumo diário.
Palavras-chave: Sal gourmet, *Eichhornia crassipes*, Composição Química, EDX

1 INTRODUCTION

Historically, there is evidence of salt production and transport in Asia since at least 2,000 b.C. in the Zhongba region in China, as stated by Zhu (2005).

At the beginning of the 21st century, the dietary norms of several countries recommendedthe daily ingestion of 6.0 grams of salt per individual, an amount that can be easily exceeded without a balanced diet (Dahl, 1958). Meanwhile, the World Health Organization recommends that adults consume less than 2,000 mg of sodium, equivalent to 5.0 grams of salt per day (WHO, 2016).

Selmera and Kristiansen (2000) claimed that the majority of the population believes that the salt found in food causes health problems and, thus, tends to reduceits consumption. As a consequence, their demand decreases, not only for salt but also for salt-preserved products.

Currently, the salt sold in Brazil continues to be produced, as established by the National Health Surveillance Agency (ANVISA), under Resolution RDC #23 of 04/24/2013 (BRASIL, 2013). The concentration of iodine is requiredto range between 15 and 45 mg/Kg of the product.

Gourmet salts vary considerably in flavor, chemical composition, and color. There is significant variability in the types of gourmet salts depending on their location of extraction, production, and, consequently, chemical composition.

Considering the vast number of gourmet salts available for consumption, it should be noted that each salt has a unique characteristic, such as sodium content or any other chemical specificity, thus contributing to the well-being of the digestive tract. Therefore, there is a need to chemically evaluate these compounds to complement the data present in the literature.

The aim ofthe presentstudywas to compare the chemical composition of different types of gourmet salts and the salt obtained from the floating aquatic macrophyte *Eichhornia crassipes*, also known as "water hyacinth", whose final product is the well-known "indigenous salt".

2 THEORETICAL REFERENCES

The U.S. Food and Drug Administration (FDA, 2016) classifies salts into: i) table salt; ii) sea salt; iii) kosher salt; and, iv) rock salt, three of which are primarily for human consumption. The agency requires that their composition be of 97.5% NaCl (sodium chloride) and 2.5% other minerals, that is, chemical compounds generated in the production process.

Currently, several types of salts can be found in food markets and storesspecialized in condiment products, including: 1) Table salt or refined salt(400mg of sodium/g of salt) (MOTA, n.d.), which can be iodinated or not, iodine being added in cases of hyperthyroidism or goiter (KNOBEL and MEDEIROS-NETO, 2004); 2) Hypo-sodium or lite salt (197 mg of sodium/g of salt), which, according to Anvisa (ANVISA, 2000), is made from mixtures of salts, whose maximum sodium content is 50%, in the same amount of NaCl. The consumption of this low-sodium salt should be overseen by a doctor or nutritionist; 3) Liquid salt (110 mg of sodium/g of salt), obtained by dissolving high-purity salt without additives in mineral water, is used in all foods, without changing their characteristics (MOLINA *et al*., 2003); 4) Sea salt (420 mg of sodium/g of salt), which is obtained by the evaporation of evaporation ponds, thus preserving its mineral content (SILVA, 2007); 5) Pink Himalayan salt (230 mg of sodium/g of salt), which contains more than 80 minerals, such as calcium, magnesium, potassium, copper, and iron (ELPAIS, 2018); 6) Black salt (380 mg of sodium/g of salt), also known as "*Kala Namak*", which is extracted in central India in natural reserves. In addition to its dark pinkish-gray color, this salt contains sulfuric compounds, iron, and other micronutrients (CARREIRA, 2014); 7) Kosher salt,a coarse salt processed into smaller, non-iodized crystals (BAAS-BECKING, 1931), and 8) Rock salt, a coarse salt that still contains inedible impurities. It is produced in three ways: from underground mining in "salt mines", solution mining, or solar evaporation (SILVA *et al*., 2000).

Some indigenous peoples manufacture salt in small quantities from the ashes of the floating aquatic macrophyte[1] *Eichhornia crassipes*, commonly known as "water hyacinth" (MELATTI, 1993).

According to Reis (2012), who studied the chemical composition of *Eichhornia crassipes* from the Almada river in Bahia, Brazil, macrophytes have high phenotypic plasticity, with the ability to rapidly increase in biomass as a function of the environment due tothe high levels ofnitrogen and phosphorus, but with adaptations that enable development even in oligotrophic environments[2]. The author analyzed some chemical parameters after harvest in February 2012, and their variations regarding dry matter (DM) were as follows: a) Organic matter (83.30 to 87.70% DM); b) Ashes (12.30 to 16.70% DM); c) Carbon (24.90 to 37.70% DM); d) Nitrogen (0.88 to 2.36% DM); e) Crude protein (5.50 to 14.75% DM), and f) Phosphorus (0.03 to 0.10% DM).

Henry-Silva and Camargo (2006), who evaluatedthe chemical composition of floating aquatic macrophytes used in agricultural effluent treatment, found that the contents were similar to those reported by Reis (2012): a) Mineral matter (17.09%); b) Nitrogen (2.0%); c) Crude protein (12.45%), and d) Phosphorus (0.26%).

The floating aquatic macrophyte *Eichhornia crassipes* is a monocot that belongs to the family *Pontederiaceae*, a small family with wide pantropical distribution, including approximately 10 genera and around 30 species. In Brazil, 5 genera and about 20 species have been reported, according to Hoehne (1979) and Souza and Lorenzi (2005).

Currently, the most used techniques in the evaluation of these inorganic chemical compounds includeAtomic Absorption Spectrometry (AAS), Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES), Optical Emission

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[1]Aquatic plants are plants that have adapted to living in aquatic environments (saltwater or freshwater). They are also referred to as hydrophytes or macrophytes to distinguish them from algae and other microphytes. A macrophyte is a plant that grows in or near water and is either emergent, submergent, or floating. In lakes and rivers, macrophytes provide cover for fish, substrate for aquatic invertebrates, produce oxygen, and act as a food source for some fish and wildlife (EPA, 2020)

[2] Characterized by the low accumulation of dissolved nutrient salts, supporting onlythe sparse growth of algae and other organisms, and having high oxygen content owing to the low organic content.

Spectrometry (OES), X-Ray Diffraction (XRD), and X-Ray Fluorescence (XRF). Although thelatter two have the same principle, that is, use an X-ray source, directly affecting the sample, several phenomena occur,leading to the attenuation of the X-ray beam, both by absorption and by scattering (SOUSA, 2011).

Thus, according to Albers *et al*. (2002), for a more specific chemical evaluation, the XRD technique qualitativelydetermines which crystalline species is present in a given sample, such as Halite (mineral NaCl), for example, using the most intense and characteristic Muller indices of each inorganic chemical species. While the EDX technique provides quantitative information on sample content (X% Na and Y% Cl) by comparison with appropriate standards (PINTO, 2013), the quantification of species by XRD is difficult to obtain due to the height of the peaks, which depend not only on quantity but also the degree of crystallinity of the sample.

Energy-Dispersive X-ray spectrometry (EDX) is the most widely used analytical technique for the elemental characterization of inorganic samples. More specifically, the method is based on the irradiationof a sample by an X-ray beam, in which the atoms present in the sample generate characteristic rays, known as “fluorescent” X-rays, that have specific wavelengths and energy that are unique regarding each chemical element. Qualitative analysis can be done by investigating the X-ray wavelengths, whereas quantitative analysis assesses the number of X-rays exhibiting the specific wavelength of each chemical element, since the intensity of fluorescent X-rays is a function of the chemical concentration of the species in the sample (PARREIRA, 2006).

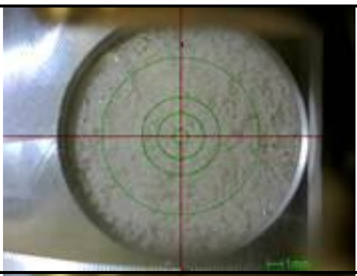
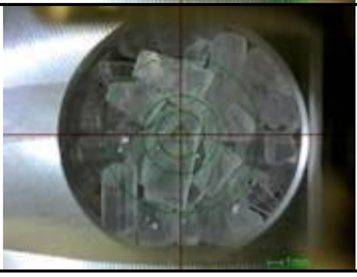
3 MATERIAL AND METHODS

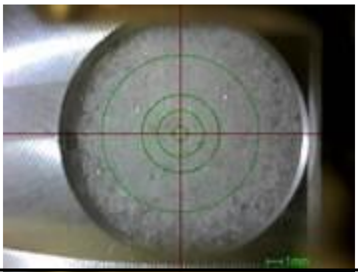
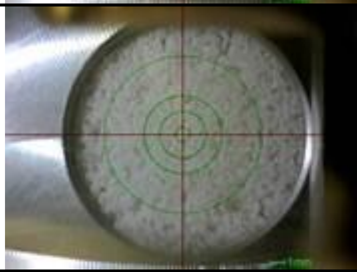
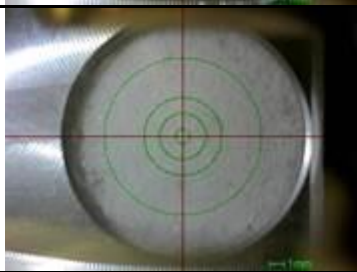
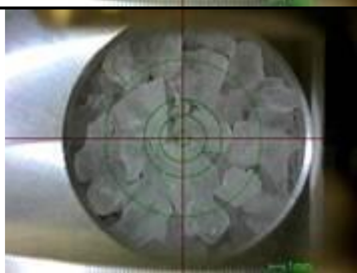
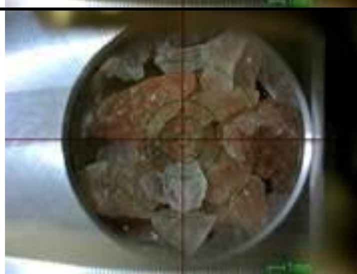
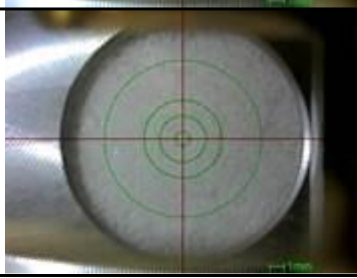
Samples of gourmet salts were purchasedfrom different commercial establishments, such as supermarkets and stores specialized in condiment products. The sample of indigenous salt was obtained from the floating aquatic macrophyte known as “water hyacinth” (*Eichhornia crassipes*), which, after harvesting, was washed, dried, and burned. The plant wasidentified and collected in an Aweti village, in Xingu Park, located in northern of state Mato Grosso, Brazil.

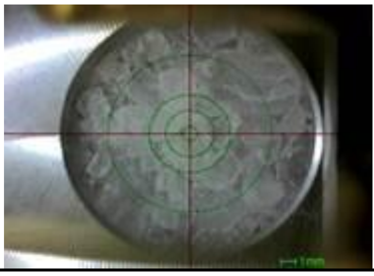
After burning, the ashes were dissolved in water and filtered to remove impurities. Following filtration, the residual saline solution was evaporated, producing salt with high KCl content, that is, potassium chloride.

The samples, shown in Table 1, were analyzed by energy-dispersive X-ray spectrometry (EDX), using a Shimadzu 7000 device.

Table 1 - List of samples and photos with the respective masses used for quantification

Sample number	**Sample name**	**Sample photo**	**Sample mass (g)**
1	Indigenous Salt (Brazil)		4.38
2	Black Salt (South Africa)		4.39
3	Himalayan Black Salt (Pakistan)		4.36
4	Himalayan Halite Salt (Pakistan)		4.33

5	Persian Blue Salt (Iran)		4.33
6	White salt (India)		4.36
7	Sicilian white salt (Italy)		4.34
8	HawaiianBlack Salt (United States)		4.33
9	Desert Salt (South Africa)		4.34
10	PinkHimalayan Salt (Pakistan)		4.35
11	Salt Flower (France)		4.34

12	Maldon salt (England)		4.34

Source: The authors

In order to determine the chemical species present in the salts, a collimator was used to select a circular region measuring 10mm in diameter. The excitation and detection of the characteristic rays were conductedin an air atmosphere, under low pressure, quantifying the chemical elements in the Al-U range (electrical voltage and current of 50kV and 17μA, respectively) and Na-Sc (15kV of electrical voltage and electric current of 310μA).

The equipment was calibrated to quantify the macro elements Cl and Na since they represent more than 80-90% of the samples' mineral content.

The measurement time was between 3 and 4 minutes, including the live time and dead time (adjusted from 10 to 20%) for each sample.

The EDX spectra was obtained using samples *in natura*, standardizing the masses to 4.3X, numbered from 1 to 12.

4 RESULTS AND DISCUSSION

The analyzed samples of gourmet salts were quantified in their mineral portions, and the results show the percentage of each chemical element detected. They were divided by region for better visualization and interpretation, that is, in salts of easternorigin (Table 2) and of western origin (Table 3).

Elements that went undetectedin the samples may have been present at quantities below the equipment's detection limit or may not have been present at all.

The Energy dispersive X-ray fluorescence (EDXRF) quantification of indigenous salt, shown in Figure 1, revealed high levels of chloride (35.834%) and potassium (62.146%), followed by sulfur (1.095%), calcium (0.450%), and other chemical elements in smaller quantities, such as bromine (0.177%), silicon (0.124%), magnesium (0.123%), copper (0.029%), and rubidium (0.023%).

Figure 1 - Measurement and quantification conditions of Indigenous Salt (Brazil)by EDX

Measurement Condition

Instrument: EDX-7000 Atmosphere: Vac. Collimator: 10(mm)

Analyte	TG	kV	uA	FI	Acq.(keV)	Anal.(keV)	Time(sec)	DT(%)
Ti-U	Rh	50	60-Auto	----	0 - 40	0.00-40.00	Live- 100	41
Na-Sc	Rh	15	187-Auto	----	0 - 20	0.00- 4.40	Live- 100	40

Quantitative Result

Analyte	Result	[3-sigma]	Proc.-Calc.	Line	Int.(cps/uA)
K	62.146 %	[0.118]	Quan-FP	K Ka	134.2663
Cl	35.834 %	[0.048]	Quan-FP	ClKa	267.2107
S	1.095 %	[0.006]	Quan-FP	S Ka	16.3111
Ca	0.450 %	[0.056]	Quan-FP	CaKa	0.6893
Br	0.177 %	[0.001]	Quan-FP	BrKa	18.2295
Si	0.124 %	[0.005]	Quan-FP	SiKa	0.5427
Mg	0.123 %	[0.029]	Quan-FP	MgKa	0.0649
Cu	0.029 %	[0.002]	Quan-FP	CuKa	1.4224
Rb	0.023 %	[0.001]	Quan-FP	RbKa	2.7844

Source: The authors

Since this evaluation technique is multi-elementary, the chemical elements may have been forming combinations of distinct types of salts from their different negative ions (Cl^-, S^{2-}, Br^-) and positive ions (K^+, Ca^{2+}, Si^{4+}, Cu^{2+}, Mg^{2+}, Rb^+), such as a) Potassium Chloride (KCl); b) Calcium Chloride ($CaCl_2$); c) Magnesium chloride ($MgCl_2$), and so on.

Another point that deserves attentionwas the absence of the element Iron (Fe) in the sample, an essential macronutrient inplant development.According to Henry-Silva and Camargo (2006) and Branco (1986), who analyzed the chemical properties of *Eichhornia crassipes*, the enrichment of waters with nitrogen and phosphorus may cause iron content to become limited.

The absence of iron in the sample can be explaineddue to its possible precipitation during the solubilization process of the ash, rendering the medium basic. In other words, the element (Fe) in alkaline medium precipitates in the form of iron oxide and hydroxide (OLIVEIRA *et al*., 2013) as hematite, magnetite, and goethite minerals, being removed from the system due to the filtration of the saline solution.

In Table 2, a predominance of Chloride and Sodiumcan be observed. few differences between the eastern salts were found, with the exception of the Black

Himalayan Salt (Pakistan), which exhibited high sulfur (S) content, followed by the element potassium (K); and the Persian Blue Salt, which presented greater chloride, sodium, and potassium content than the other chemical groups.

Table 2 - Multi-elemental quantification of the chemical composition of eastern salt samples in comparison withthe indigenous salt

Chemical species (%)	Indigenous (Brazil) (1)(*)	Himalayan Black (Pakistan) (3)(*)	Himalayan Halite (Pakistan) (4)(*)	Persian Blue (Iran) (5)(*)	White (India) (6)(*)	Pink Himalayan (Pakistan) (10)(*)
Cl	35.834	72.570	75.307	67.056	79.319	76.354
Na	---	19.182	22.521	16.345	18.327	18.585
S	1.095	5.133	0.616	0.207	0.242	0.664
K	62.146	1.066	0.452	15.128	0.377	1.562
Ca	0.450	0.929	0.373	0.953	0.831	0.872
Fe	---	0.374	---	---	0.050	0.217
Br	0.177	---	0.021	0.195	0.086	0.021
Si	0.124	0.071	0.033	---	0.247	0.708
Cu	0.029	0.058	0.050	0.032	0.042	0.046
Sr	---	0.009	0.008	0.017	0.020	0.009
Mn	---	---	0.024	---	---	0.040
P	---	0.300	---	---	---	---
Pm	---	0.162	---	---	---	---
Mg	0.123	---	0.595	---	0.343	0.850
Ti	---	0.047	---	---	---	0.034
Zn	---	0.020	---	0.010	---	---
Ag	---	0.013	---	---	---	0.039
Ir	---	0.034	---	---	---	---
Rb	0.023	0.005	---	---	0.003	---
Br	---	0.026	---	---	---	---
Sc	---	---	---	0.035	---	---
Po	---	---	---	0.022	---	---
Gd	---	---	---	---	0.047	---
Tb	---	---	---	---	0.047	---
Os	---	---	---	---	0.019	---

Legend: (---) There was not enough excitation of the element for quantification/the element was not present.
(*) These numbers refer to the matrix analysis of the respective salts shown in Table 1.
Source: The authors

It can be noted that most of the salts exhibited a high degree of purity, particularlyregarding Chloride, Sodium, Potassium, and Sulfur, with the sum of the percentage of these elementsreaching approximately 98%. Theindigenous saltpresented 97.98% purity,considering chloride and potassium alone.

Table 3 shows a prevalence of Chloride and Sodium, with little difference between the western salts, with the exception of the South African Black Salt, which exhibitedhigh sulfur content (2.008%), followed by chloride, sodium, and potassium,when compared to the othersalts.

Table 3 - Multi-elemental quantification of the atomic chemical composition of western salt samples in comparison with the indigenous salt.

Chemical species (%)	Indigenous (Brazil) (1) (**)	Black(*) (South A.) (2) (**)	Sicilian white (Italy) (7) (**)	Hawaiian Black (USA) (8) (**)	Desert (South Africa) (9) (**)	Salt Flower (France) (11) (**)	Maldon (England) (12) (**)
Cl	35.834	75.803	79.319	81.038	76.800	79.130	81.445
Na	---	20.035	18.327	15.514	20.045	19.798	17.325
S	1.095	2.008	0.242	0.518	0.941	0.097	0.068
K	62.146	0.756	0.377	0.775	0.429	0.234	0.121
Ca	0.450	0.534	0.831	0.688	0.019	0.533	0.406
Fe	---	0.206	0.050	0.036	0.006	0.023	0.014
Br	0.177	---	0.086	0.113	---	0.054	0.040
Si	0.124	0.078	0.247	0.023	0.038	---	---
Cu	0.029	0.031	0.042	0.047	---	0.043	0.031
Sr	---	0.010	0.020	0.008	---	0.011	0.008
Mn	---	---	---	---	---	---	0.015
P	---	0.201	---	---	---	---	---
Mg	0.123	---	0.343	1.228	---	---	---
Zn	---	---	---	0.012	---	0.010	0.031
Ag	---	---	---	---	0.040	---	0.023
Rb	0.023	---	0.003	---	---	0.002	0.002
Pd	---	0.062	---	---	---	---	---
Hf	---	0.058	---	---	---	---	---
Cr	---	0.082	---	---	---	---	0.081
Hg	---	0.029	---	---	---	---	---
Ni	---	0.028	---	---	---	---	---
Br	---	0.025	---	---	0.041	---	---
Tl	---	0.019	---	---	---	---	---
Se	---	0.018	---	---	---	---	---
Ga	---	0.014	---	---	---	---	---
Pa	---	0.003	---	---	---	---	---
Gd	---	---	0.047	---	---	---	---
Tb	---	---	0.047	---	---	0.037	---
Os	---	---	0.019	---	---	---	---
Nd	---	---	---	---	0.626	---	0.299
Pr	---	---	---	---	0.598	---	---
V	---	---	---	---	0.345	---	---
Pt	---	---	---	---	0.045	---	---
Au	---	---	---	---	0.014	---	---
Th	---	---	---	---	0.007	---	---
At	---	---	---	---	0.007	---	---
Hf	---	---	---	---	---	0.023	---
Co	---	---	---	---	---	0.004	---
W	---	---	---	---	---	---	0.092

Legend: (---) There was not enough excitation of the element for quantification/the element was not present.
(*) Including South A.
(**) The numbers refer to the matrix analysis of the respective salts shown in Table 1.
Source: The authors

It can also be noted that most of the salts presented a high degree of purity regarding chloride and sodium, while the indigenous salt was comprised primarily of chloride and potassium.

Regarding the quantified salts, with the exception of the indigenous salt, their elementary matrix was basically composed of Cl and Na.

The mean composition of the 11 samples (except the indigenous salt) of gourmet salts was 76.740% and 18.728% of Cl and Na, respectively, with a standard deviation of 76 ± 4 for Cl and 19 ± 2 for Na. All samples were within this static range, with the exception of the Hawaiian Black Salt (Hawaii) considering the elements Cl and Na, and Maldon Salt (England) regarding the element Cl.

The trace elements highlighted in Tables 2 and 3 were present in most samples, as part of mineral impurities and/or derived from the production process.

There has been a worldwide trend for the reduction of sodium in food (CDC, 2010), especially in industrialized sausages (STROM *et al*., 2013), due to its relationship with high blood pressure, coronary diseases, and stroke.

In general, several indicationslinking the elevated consumption of NaCl, that is, sodium chloride, to the development of chronic diseases can be found in the literature (HE and MACGREGOR, 2007).

According to the obtained results, it is worth mentioning that this indigenous salt should be consumed under medical guidance due to its high concentration of potassium chloride, a salt with specific properties for human treatment and not daily consumption.

The study by Lawson (1981), conducted with special patients, confirms the concomitant presence of hypokalemia[3], reinforcing the strict need for its monitoring and the replenishment of potassium, when necessary.

[3] Hypokalemia, according Soar *et al*., 2010, is defined as the low level of potassium (K^+) in the blood serum.

5 CONCLUSIONS

The energy-dispersive X-ray spectrometry technique proved to be an adequate tool in the elementary evaluation of different salts used by the human population.

Regarding the quantified salts, with the exception of the indigenous salt, their elementary matrix is basically composed of chloride and sodium. Unlike the other samples, the elementary matrix of indigenous salt is comprised of chloride and potassium.

More than 40 trace elements were detected and quantified in reduced quantities in the samples, with considerable variability in quantity and diversity.

In most samples, the trace elements detected pertained to mineral impurities and/or were derivedfrom the production process.

The indigenous salt should be used under medical guidance, except in traditional indigenous populations that produce and consume the salt, thus preserving the culture and its traditions.

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