

Original Paper

Inorganic Chemical Composition Analysis of 10 Different Types of Commercial Salt by Wavelength Dispersive X-Ray Fluorescence (WDXRF) Method

Anne B. Bastos¹, Hugo R. A. Carvalho¹, Cláudia C. Silva^{1*} & Regina Helena de Almeida Santos²

¹ Grupo Crowfoot de Métodos de Raios-X, Escola Superior de Tecnologia, Universidade do Estado do Amazonas, UEA. Av. Darcy Vargas, Manaus, AM, Brazil

² Instituto de Química de São Carlos, Universidade de São Paulo. Avenida Trabalhador São-carlense, São Carlos, SP, Brasil

* Cláudia C. Silva, Grupo Crowfoot de Métodos de Raios-X, Escola Superior de Tecnologia, Universidade do Estado do Amazonas, UEA. Av. Darcy Vargas, 1200, CEP 69065-020, Manaus, AM, Brazil

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Abstract

Salt is one of the most used seasoning in culinary with a great variety of them. Despite that, there is not a lot of published studies that analyses its compositions, differences and similarities between them. Thus, this research aims to determine the inorganic composition of table, light, pink and black Himalayan, Hawaii's red, Persian blue, Mediterranean sea and three Argentinian gourmet salts by the Wavelength Dispersive X-ray Fluorescence (WDXRF) method in order to compare them using PCA and HCA analysis. Na and Cl were major elements found in all samples. As for trace elements: Al, Si, S and K in drastically different concentrations, due to conservation of nutrients from the soil, water or additives. These differences were used to distinct three groups showing that there is between 70 and 60% similarity among the nine samples, while the light salt does not have similarity to any other salt studied.

Keywords

salt, inorganic composition, X-ray fluorescence, PCA, HCA

1. Introduction

Halite, the geological name of NaCl, can be found in two forms: sea salt and gem salt, also known as rock salt. The evaporation process of seawater provides the sea salt. While the rock one is salt extracted from underground mines that have already been submerged by seawater forming a saline deposit when they dry out (Cornelis, 2010).

One of the differences between the salts is in their production process, which the most commonly used are: underground or solution mining (De Melo, De Carvalho, & De Carvalho Pinto, 2008). Since seawater contains several dissolved salts, these are precipitated and separated from the final product. There is also the addition of iodine and anticaking agents to the product. Table salts are usually produced in this way (Aditivos & Ingredientes, 2016; Da-Col, Bueno, & Melquiades, 2015).

Besides production process and color, composition is also another factor that differ the types of salt, because it varies in sodium and mineral concentration. Although there is usually a need to remove compounds that may affect the product, some rock salts does not require the refining process due to high purity, such as the Himalayan pink and black salt and the Persia blue salt (De Melo, De Carvalho, & De Carvalho Pinto, 2008).

Himalayan pink salt is a rock salt extracted from the Khewra mines, located in the Jhelum district of Punjab city (Pakistan). This mine is the second largest in the world and salt deposition occurred mainly in the Precambrian period. The pink salt has trace elements, such as iron, calcium and magnesium, from the rich mineral composition of the soil; and it has several colorations: white, pink and reddish, which vary according to the extraction site and ore composition (Rahman, Islam, & Farrukh, 2015).

The black salt contains volcanic halite that have traces of sodium sulfate and iron sulfide, the last one responsible for a slightly bitter taste. The most notable feature is the extremely strong smell due to hydrogen sulfide found in the groundwater, salt mines, sulfide ore and volcano emissions that impregnates in the salt (Kalra, S., Kalra, B., & Sawhney, 2013; Oliveira, Miranda, Silva, Batista, Menezes, Dantas, & Farias, 2013).

Iran, ancient Persia, provides the Persian blue salt. This country presents mines in several locations that store halite formed in Cenozoic era. The Kavir mines are one of those which is formed by rock salt with the presence of minerals, mainly clay constituents (Folle, 2007; Geluk, 2000).

Some types of salts have a different composition due to the spices in them added. These are known as gourmet salts and, depending on the additive, have a distinct taste and color. The Argentine brand Shio® specializes in the production of those that include salt with smoked peppers and vanilla; with truffles and a mixture of spices called Ras el hanout; and with malbec wine (Shio®, 2017).

There are also sea salts that follow the example of rock ones and maintain their nutrients, receiving none of the refining processes. The Hawaii's red salt and the Mediterranean Sea salt are some examples.

Hawaii's red salt is a reddish-colored salt extracted by the evaporation of the Hawaiian Islands waters. This characteristic color is caused by adding Alaea, a volcanic origin clay mineral that has a high

concentration of iron oxide, which guarantees the reddish color (Drake, S. L. & Drake, M. A., 2011). The Mediterranean Sea salt, as the name suggests, comes from the waters of the Mediterranean, white in color. Since the Tertiary period, in the region of extraction, there is a large accumulation of evaporites, which supposedly will lead to a salt rich in minerals (Goffredo & Dubinsky, 2014; Mohriak, Szatmari, & Anjos, 2009). However, Stergiou (2016) report that the Mediterranean salt has a high level of purity and does not contain other elements, besides Na and Cl, in significant concentrations (Stergiou, Karageorgiou, Theodoridou, Giouri, Papadopoulou, & Melfos, 2016).

Despite the variety of salts, the current concern is with the consume reduction of the condiment. In recent years, excessive salt consumption has been associated with the increase in the number of chronic diseases, such as hypertension, cardiovascular and renal diseases, osteoporosis, among others. The search for alternatives to replace the table salt has become a necessity (Nilson, Jaime, & Resende, 2012).

Light salt is a product with a reduced sodium content. According to the Agência Nacional de Vigilância Sanitária - ANVISA (1995), the mixture of sodium chloride with other salts should result in a product similar to the common one with the same salting power, but with a maximum content of 50% sodium in the same amount of NaCl (Agência Nacional De Vigilância Sanitária (ANVISA), 2016).

Thus, given the wide variety of salts, a study that is able to determine the chemical composition, its characteristics and whether there are significant differences between them is necessary.

2. Experimental

2.1 Samples

The samples were bought in local markets. Professor Regina Helena de Almeida Santos from Universidade do Estado de São Paulo (USP) gave the pink salt. While Prof. Cláudia Cândida Silva acquired the black, blue, red, Mediterranean and gourmet ones.

The Table 1 shows the nomenclature used for each sample.

Table 1. Samples' Nomenclature

Sample	Nomenclature	Sample	Nomenclature
Common salt	CS	Mediterranean Sea salt	MS
Himalayan pink salt	PS	Malbec wine salt	WS
Himalayan black salt	BS	Truffle salt	TS
Hawaii's red salt	RS	Vanilla salt	VS
Persian blue salt	PBS	Light salt	LS

Three samples of each salt were prepared, obtaining 30 samples in total. The salts were crushed using agate mortar and pestle, followed by weighing them in an analytical balance in the ratio of 1: 4; that is, 1

g of sample to 4g of boric acid (H_3BO_3) which was used as agglutinator element.

The gourmet salts received an additional process to eliminate the species. It was possible to use a simple sieve to separate salt and its additives. With the salt isolated, the same procedures already described were carried out.

The pellets preparation used a pneumatic press for the agglutination of the sample in the boric acid.

2.2 Wavelength Dispersive X-Ray Fluorescence (WDXRF)

Wavelength Dispersive X-Ray Fluorescence method analyzed the pallets sample composition, qualitative and quantitative analyses, using the Rigaku equipment, Supermini model, with analysis of elements with atomic number between 11 and 92 (Perring & Andrey, 2003; Pereira, Pereira-Filho, & Bueno, 2006).

All data were obtained using a Rh X-ray tube, a set of analyzer crystals (LiF, PET, and RX25), time of 250 s of exposure and with a voltage of 40 keV. The tube current was 40 mA and its voltage was 50 kV for heavy metals, Sc, Ca, K, S, P, Si, Al, Mg, Na and F. All measurements were carried out in vacuum and the readings occurred during intermittent days (Leyden, 1984).

The characteristic X-ray radiation of $K\alpha$ -line and the background radiation were measured for the determination of each element, while the concentration were based on their relative intensities (cps/uA) using external standards (Araújo, Conceição, Barbosa, Teresa Lopes, & Humanes, 2003). The spectral profile of WDXRF for one of the blue salt samples, represented in the Figure 1 below, shows the elements found and its intensities.

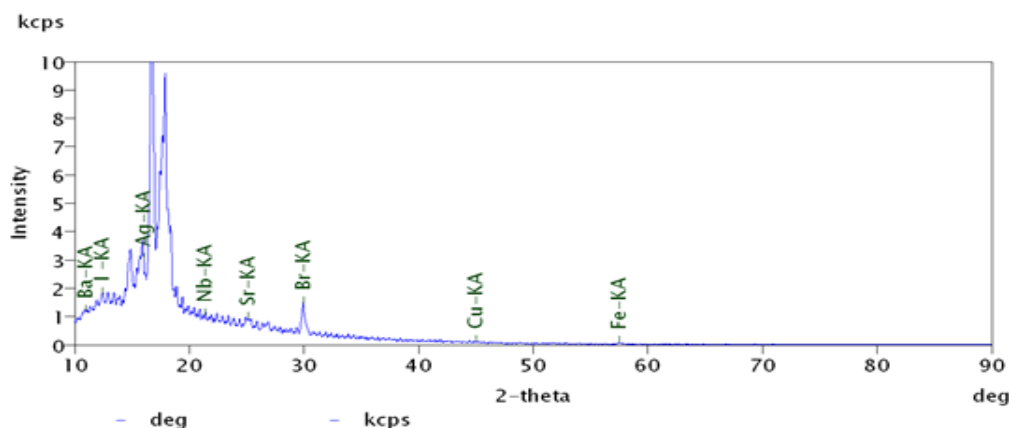


Figure 1. Persian Blue Salt Spectral Profile of WDXRF for Heavy Metals

Geological reference materials: such as GBW 3125, GBW 7105, 7113 GBW were employed in the equipment calibration, and also for determining the accuracy and precision thereof. Thus, the average error ranging between 1 and 12% for the elements quantified here were obtained. Aiming to measure accuracy and precision of the method, the same patterns were analyzed five times each, reaching an average standard deviation of 7%. The detection limits were calculated using the formula suggested by Araújo et al. (2003). Reaching values of 10 ppm for the heavier elements and 150 ppm for the lower

atomic number. The intensities found in the first analysis of the samples allow us to decide that sample dilution of 1:7 boric acid would be enough to compensate the matrix effect.

Salts of known purity of Al, Si, Ca, S, K, Fe and Cu, diluted in boric acid, also of known purity, were used in six predetermined concentrations, which were subjected to the same analysis conditions as the samples. These patterns were analyzed under the same conditions as the samples, generating standard curves that were used for quantification of each element (Bertin, 1975).

2.3 Chemometrics Analysis

Through the Pirouette program (InfoMetrix, Woodinville, Washington, USA) version 4.0, PCA (Principal Component Analysis) and HCA (Hierarchical Cluster Analysis) methods were used as chemometric analyzes.

The number of parameters used in analyzes of this type is usually high, but is also adequate for a fewer number. In order to verify the similarity between the samples, the PCA and HCA methods are useful because they do not take into account any information regarding the identity of the samples and allow the graphic visualization of the entire dataset, examining the presence or absence of clusters between the samples (Scapin, Salvador, Lima, Scapin, & Prestes, 2002).

With the mean results in triplicate of the salts, resulting in 10 samples, and using the concentration of the 22 chemical elements found in them, a 10x22 data matrix was centered on the mean. This initial treatment is used when all the variables are in the same unit, having the same magnitude, as usually occurs in spectroscopy. This way, noises do not interfere in the analysis (Ferreira, 2015).

The HCA dendrogram used Euclidean distance and complete linkage approach. This method has the tendency to form more compact clusters, since the calculation of the distance between the groups is based on the greater dissimilarity between the parameters. Although, it is more sensitive to anomalous samples, because it uses the greater distance between the objects to define the distance between the groups (Matos, Pereira-Filho, Poppi, & Arruda, 2003).

3. Results and Discussion

The Table 2 shows the mean results of WDXRF qualitative and quantitative analysis.

Table 2. Qualitative and Quantitative Mean Results of Salt Samples

Elements	Samples concentration in % mass									
	CS	LS	PS	BS	PBS	RS	MS	VS	TS	WS
Na	65.9560	26.1916	63.3539	60.5652	57.2773	51.8087	63.0658	61.8574	64.4900	60.3000
Mg	-	-	0.6279	1.3150	-	-	-	-	-	-
Al	0.0625	0.0619	0.1014	0.6803	0.0588	0.0841	0.0478	0.0637	0.0558	0.0469
Si	0.0318	3.8846	0.1990	1.9855	0.0511	0.0893	0.0274	0.0837	0.0393	0.0355
P	0.0154	-	-	0.1769	-	-	-	0.0197	0.1062	0.0207

S	0.0291	0.0374	0.7295	0.6832	0.4984	0.0985	0.1340	0.0484	11.7337	0.2363
Cl	33.8486	44.8621	34.3939	32.2919	39.0421	35.0084	36.5938	37.7729	23.3606	39.1072
K	0.0526	24.7563	0.2884	0.4851	2.5938	0.0710	0.0477	0.0710	0.0825	0.0560
Ca	0.0189	-	0.1552	1.6053	0.3511	0.0613	0.0837	0.0429	0.0668	0.1428
Fe	0.0113	-	0.0126	0.1449	0.0120	0.1498	0.0076	0.0112	0.0082	0.0070
Br	0.0079	0.0303	0.0055	0.0058	0.0199	0.0070	0.0061	-	0.0056	-
Rb	-	0.0095	0.0044	-	-	-	0.0020	-	0.0044	0.0047
Rh	-	0.1582	-	-	-	-	-	-	0.0519	-
Ag	-	0.0494	-	0.0533	0.0579	0.0203	0.0152	0.0574	0.0197	0.0353
Sr	-	0.0180	-	0.0074	0.0058	-	0.0021	-	0.0032	0.0034
Y	-	0.0134	-	-	-	-	0.0026	-	0.0036	0.0047
I	0.0108	-	-	-	0.0258	-	-	-	-	-
Ba	-	-	-	-	0.0405	-	-	-	-	-
Nb	-	-	-	-	0.0052	-	-	-	-	0.0034
Mo	-	-	-	-	-	-	-	-	0.0042	0.0050
Cd	-	-	0.0280	-	-	-	-	-	0.0084	0.0467
Mn	-	-	0.0043	-	-	-	-	-	-	-

The results revealed a great variety of elements with main constituents being sodium and chlorine.

Figures 2 and 3 illustrate the Na and Cl mass percentage concentrations, respectively, of each sample.

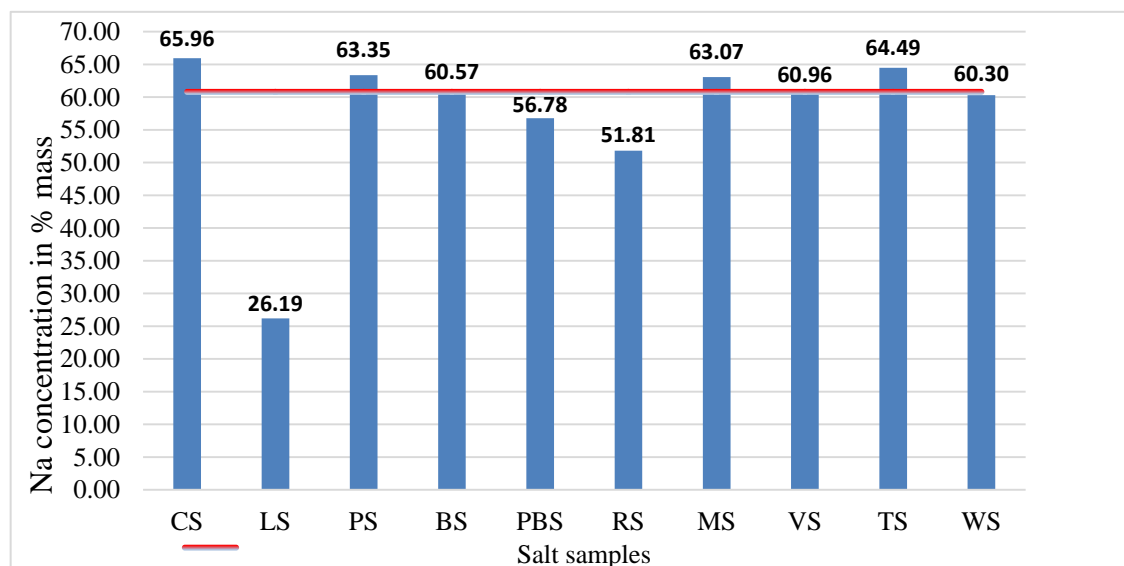


Figure 2. Sodium Concentration in Samples

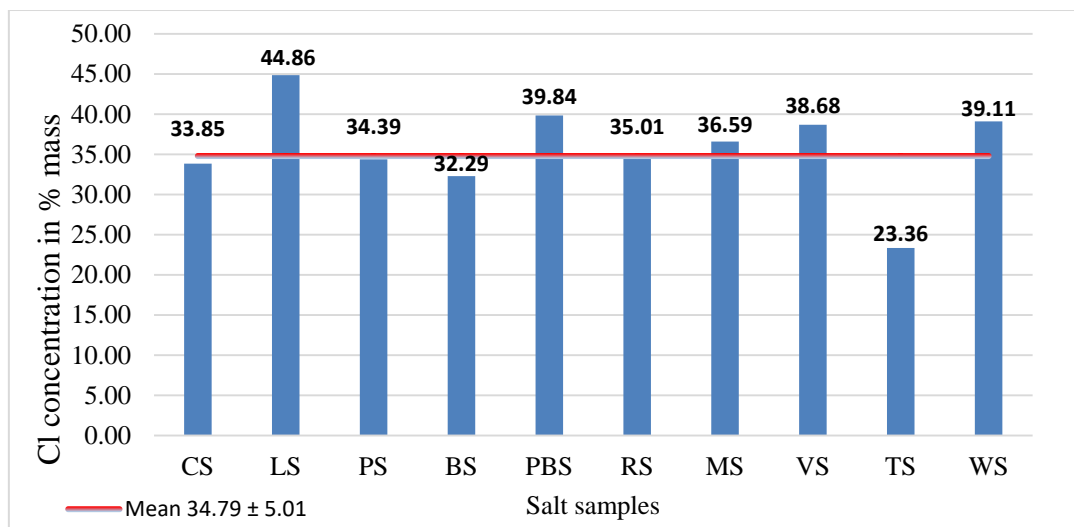


Figure 3. Chlorine Concentration in Samples

The mean and standard deviation values in the last two graphs calculated did not consider the anomalous values exhibited by the light salt samples. Most of the salts do not present great variations of Na and Cl concentrations, since they are contained within the interval defined by the mean and standard deviation.

The light salt exhibited the lowest concentration of Na due to the obligatory reduction of the element in its production, as regulated by ANVISA (2016). Other salts with lower than average concentrations were Hawaii's red salt (51.81% Na) and truffle salt (23.36% Cl). These condiments have spices in their composition that can replace Na or Cl in the halite structure.

However, since one of the objectives of this research is to verify if there are similarities between commercial salts, the focus was placed in the elements found in all samples and how similar its concentrations can be. Trace elements in samples, illustrated in Figure 4, showed variation regarding the chemical identity and its respective concentration.

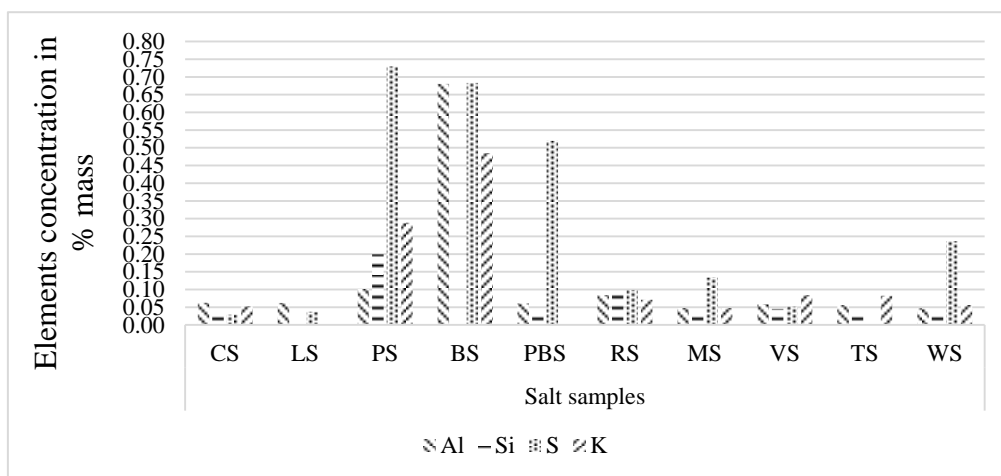


Figure 4. Percentage Mass Concentration of Trace Elements in Samples

All samples had aluminum, silicon, sulfur and potassium at concentrations lower than 0.75%. Factors such as water ionic composition; association with other sedimentary rocks, such as limestone (CaCO_3), anhydrite (CaSO_4), clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot \text{H}_2\text{O}$) and other evaporite salts; salt production techniques; and formation process of mines and salt deposits are some reasons that explain the presence and variation in the concentration of these elements (De Melo, De Carvalho, & De Carvalho Pinto, 2008).

However, some salts had peculiar concentration results for a few elements. Figure 5 shows the high percentages of potassium, sulfur and silicon removing them from the trace elements classification in some samples.

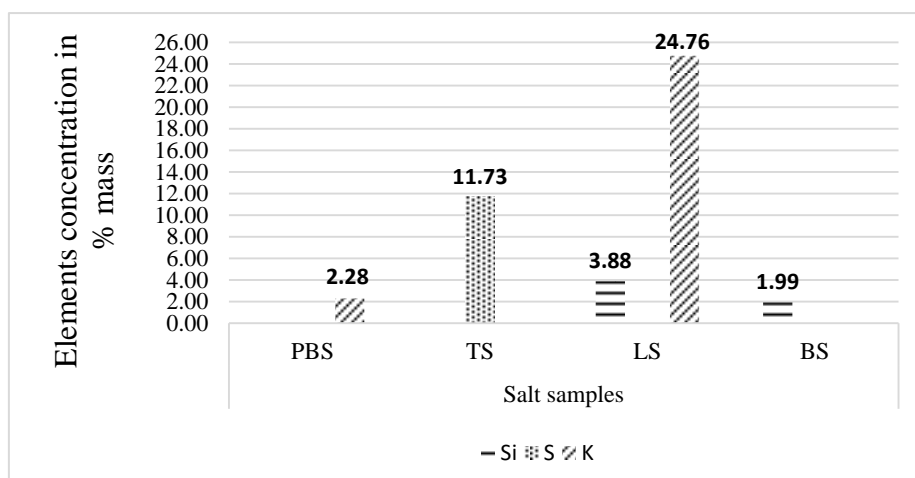


Figure 5. Samples with Peculiar Concentrations of Trace Elements

The high concentration of sulfur in TS samples relates to the presence of truffles, an underground fungus described as gourmet food due to its rarity and its characteristic flavor, texture and aroma (Rencher, 2002). Sawaya (1985) research claims that truffles have all the essential amino acids, including those with sulfur (methionine, cystine, tryptophan and lysine) that are generally the limiting factor in many whole foods (Sadler, 2003). In addition, when compared to other types of edible mushrooms, truffles have a higher protein content, justifying again the sulfur abundance (Sawaya, Al-Shalhat, Al-Sogair, & Al-Mohammad, 1985).

Light salt contains Si mostly due to the addition of substances such as aluminum sodium silicate ($\text{NaAl}_2\text{Si}_3\text{O}_8$), to reduce the air humidity absorption (Aditivos & Ingredientes, 2016). While silicon in the black salt can be associated with Himalayan soil formed by clay compound, since these natural fine-grained ore, mainly formed by hydrated aluminum silicate ($\text{Si}_2\text{O}_3\text{Al}_2(\text{OH})_4$) is found in deposits spreaded throughout the territorial portion of Pakistan and the black salt does not receive any additives or processes to eliminate impurities (Wang & Marcone, 2011).

The high concentration of K in the light salt relates to the product itself consisting in 50% sodium chloride and 50% potassium chloride. KCl is used because it has a shorter retention time in the body. Sodium chloride needs water to dissolve and act in the human body, which means the higher the intake

of common salt; the longer it will take to dissolve it, causing an overload in the circulatory system and, consequently, an increase in the blood pressure. On the other hand, potassium chloride takes less time to react in the body, reducing water retention (Shah, 1977).

In the blue salt, potassium is associated with its color. Because the Persian salt was white in color and had only a few blue crystals, an analysis performed on only these bluish grains showed a high level of potassium (7.42%) in comparison with the other three samples. Halite crystals have a wide variety of atomic defects, which act as centers of color. In Iran, the extraction region, salt forms multi-layered domes that do not always move at the same speed. Shear zones separates other parts of the salt structure where blue crystals may occur near potassium rich beds. The excess free sodium, released from minerals with potassium, whose rapid growth rate promotes the occurrence of network defects and results in a deep blue color (Teixeira, André Chaves, Diogo, Lourenço, & Menezes, 2007).

Knowing the 22 elements and its concentrations, as seen in Table 2, statistical techniques of PCA and HCA used a matrix containing all information related to the samples (arranged in rows) and with the variables (elements concentrations arranged in columns) to determine if there were significant differences between the 10 types of salts.

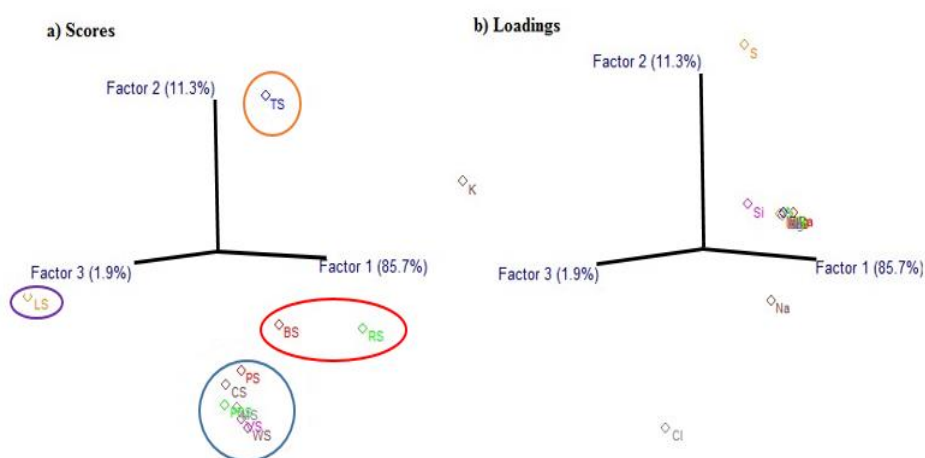


Figure 6. PCA Analysis: a) Scores and b) Loadings

The scores chart, shown in Figure 6 (a), provided discriminatory information from the samples, showing four groups. The first principal component (PC1) putted together the pink, common, blue, Mediterranean, malbec wine, and vanilla salts; as well as the red and black ones in a different group. This component models 85.7% of the information associated with the original variables. The second principal component (PC2) associated more 11.3% of the information, distinguishing the truffle salt. Finally, the third principal component (PC3) with a value of 1.9% presented information that isolated the light salt from the others.

The loadings chart in Figure 6 (b) determined the variables that influenced the samples grouping. Sodium, chlorine, potassium and sulfur concentrations were the ones that most influenced the clusters.

These, as already mentioned, are in the halite structure, extraction region composition or further added to the product. Similar Na and Cl contents approximated the eight samples through PC1, being the largest set grouped by the similarity of chlorine concentration, while the black and red salts gathered by the sodium and trace elements. On the other hand, the truffle salt was isolated because of its high sulfur concentration. While the separation of light salt was caused by the high potassium content.

In a principal component analysis where the two, or three, components have a percentage above 70% of the total variation, the plot represents the data with very little distortion. PC1 and PC2 elucidated 97% of the total available variance between the concentration parameters. Therefore, they are considered acceptable to delineate the results of variance of the data obtained from the scores and loadings graphs (Matos, Pereira-Filho, Poppi, & Arruda, 2003).

The dendrogram made by the HCA in Figure 7 confirmed the results obtained in the principal components analysis.

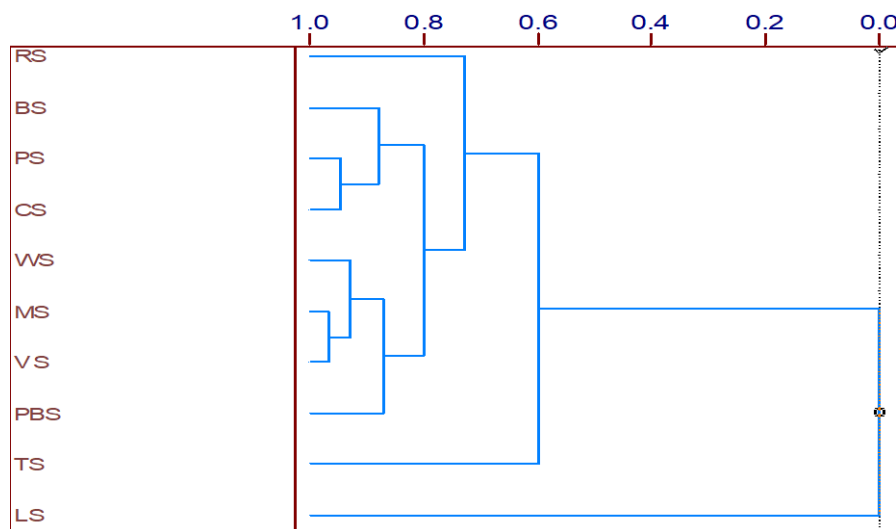


Figure 7. HCA Analysis - Dendrogram

With a similarity of more than 70%, light and truffle salts being the exceptions, the salts are very much alike. Compared with the common salt, the pink one has the highest similarity, mainly due to the sodium and chlorine concentrations.

The truffle salt has about 60% affinity with the other samples, differing by the high concentration of sulfur. It is also interesting to notice that gourmet salts, even if they are from the same region and have their spices removed, adhere so efficiently to the additives that it changes their composition.

The light salt, on the other hand, did not have similarity to any other salt studied. Low sodium content and high potassium concentration compared to the remaining nine samples were decisive criteria of separation, classifying it as an isolated salt.

4. Conclusions

Composition and concentration of elements were easily determined by the WDXRF method, which showed as major elements Na and Cl in all samples. With slightly different concentrations, the light salt was the one with lowest level of Na, which was expected, becoming the best alternative of seasoning to reduce the indigestion of sodium. The presence of K, S, Si and Al as trace components is associated with procedures taken. Maintaining the minerals present in the soil, water or extracted region provide the pink and black Himalayan, Hawaii's red, Persian blue and Mediterranean Sea salts a rich composition in these elements. The same happened with the three Argentinian gourmet salts, but they also received others additives to improve its taste that reflected in the composition. In the case of table and light salts, since they pass through process to withdraw minerals, compounds to maintain the product quality were the reason for the presence of the trace elements.

The PCA and HCA analyses identify a great similarity between the samples between 70 and 60%, while the light salt remained isolated, without resemblance with any other sample. Thus, with all the results seen, the high price or advertising about all the different types of salt may be overwhelming.

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