

## INVESTIGATION OF ELEMENTS IN SWEETENERS BY NAA ANALYTIC TECHNIQUE

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### ABSTRACT

In this study, brands of sweeteners (aspartame and sucralose) marketed in Brazil were investigated by Neutron Activation Analysis (NAA). The measurements were performed using the IEA-R1 nuclear reactor at IPEN - CNEN/SP. The elements Cl and Na were identified in all samples. The Na concentration in sweeteners samples showed low content for most of the brands (0.013 – 3.81 g kg<sup>-1</sup>), while Cl had high concentration variation (0.04 – 11.10 g kg<sup>-1</sup>). These data provide knowledge regarding nutritional aspect and toxicity derived from sweetener consumption.

### 1. INTRODUCTION

In the past two decades, LEER (Laboratório de Espectroscopia e Espectrometria das Radiações), at IPEN/CNEN-SP, has been dedicating to determine reference values in body fluids of the Brazilian population [1-9]. These analyzes require knowledge about gender, age, physical activities and eating habits among others. As a result, these studies showed that the Brazilian population makes indiscriminate use of sweeteners, aiming to reduce the excessive consumption of sugar (for reducing ingested calories) and to maintain a healthy weight.

Nowadays, the obesity related to excessive consumption of sugar has become a public health concerns worldwide [10], increasing the risk factor for many different diseases, such as cardiac conditions, hypertension, diabetes and metabolic syndrome [11-13]. To reduce the obesity, sweetener intake has increased, significantly, in the last decade: sweetener consumption in the American population (adult) increased from 27% to 41%; among children, it was 9% to 25% (an increase of more than 200%). In Brazil, there are no official statistics, but according to “Food Guide for the Brazilian Population” (Guia alimentar – ANVISA [14]), the trend is the same.

Some technological properties of sweeteners, such as flavor characteristics and caloric value, as well as physicochemical properties of synthetic and natural sweeteners have been widely

investigated, but investigations related to the inorganic elements are still scarce [15-18]. Evaluation of inorganic elements in sweeteners is important in nutritional investigations, because they may be present as constituents or contaminants. Recently, analyses of sodium content in some synthetic sweeteners have been performed [19] to verify compliance with ANVISA recommendation in relation to sodium level in processed products (tolerable intake limit < 0.4 g/kg) [20]. Now, we intend to complement this investigation. Specifically, aspartame and sucralose sweeteners were selected for this study because they have become popular, mainly in Brazil and Europe, where they are commonly used in the production of light and diet foods [15, 21, 22].

In this study, 13 brands acquired in markets of São Paulo city were analyzed by Instrumental Neutron Activation Analyses technique (INAA). The measurements were performed in the nuclear reactor at IPEN/CNEN-SP, Brazil. The evaluation of inorganic elements in sweeteners is important for nutritional investigations and for consumers.

## 2. METHODS

To perform this analysis, 13 brands of sweeteners (6 of aspartame and 7 of sucralose) marketed in Brazil were investigated. Samples were sifted and homogenized. After, each sample was weighed (80 mg) and transferred to a polyethylene tube. All the samples were prepared in triplicate. Each sample was sealed into individual polyethylene bag, together with the Certified Reference Material (CRM). Sample and Standards (CRM) were irradiated from minutes to hours, in the nuclear reactor IEA-R1, (3.5-4.5 MW, pool type, at IPEN), with a thermal neutron flux. After the irradiation, the activated samples were gamma-counted using an HPGe (Model GEM-6019), coupled to an MCA ORTEC (Model 919E). The gamma ray spectra analysis was performed using the *ATIVACÃO* in-house software. The NIST SRM 1573a and NIST SRM 1547 Peach Leaves reference materials were used as standard and for the quality control.

## 3. RESULTS AND DISCUSSION

The INAA method was validated using two Certified Reference Materials (NIST SRM 1573a and NIST SRM 1547): the accuracy of the method was evaluated by Z-score test and the repeatability by RSD values [23]. These results are presented in Table 1.

The elements Cl and Na were identified in all samples. For  $^{38}\text{Cl}$  ( $T_{1/2}\sim 37$  min,  $E_{\gamma} = 1643$  keV) determination sample and standard (NIST SRM 1547 Peach Leaves) were irradiated for 300s and, after a decay time of 120s, they were counted by 600s. For  $^{24}\text{Na}$  ( $T_{1/2}\sim 15$  h,  $E_{\gamma} = 1369$  keV) determination sample and standard (NIST 1573a Tomato Leaves) were irradiated for 300s and, after a decay time of 2h, they were counted by 3h. The Na and Cl concentrations in brands of sweeteners were presented in Table 2. The results were expressed by: Mean Value (MV), Standard Deviation ( $\pm 1\text{SD}$ ) and Minimum (min) and Maximum (max) values. Figures 1 and 2 were elaborated to show all the results. The elements Ca, Fe e S were identified in some brands, but with very low statistics.

**Table 1. The analysis of the reference materials by INAA**

Elements	Reference Material Mean $\pm$ 1SD	Present Study Mean $\pm$ 1SD	RSD, %	Z-score values
Na, mg kg <sup>-1</sup>	136 $\pm$ 4 <sup>b</sup>	135 $\pm$ 9 <sup>b</sup>	6.7 <sup>b</sup>	0.3 <sup>b</sup>
Cl, mg kg <sup>-1</sup>	312 $\pm$ 14 <sup>a</sup> *6600 <sup>b</sup>	299 $\pm$ 14 <sup>a</sup> 6560 $\pm$ 450 <sup>b</sup>	5.0 <sup>a</sup> 6.9 <sup>b</sup>	1.1 <sup>a</sup> -

<sup>a</sup> NIST SRM 1547 Peach Leaves

<sup>b</sup> NIST 1573a Tomato Leaves

MV: Mean Value

SD: Standard Deviation

RSD: Relative Standard Deviation

\* Noncertified value

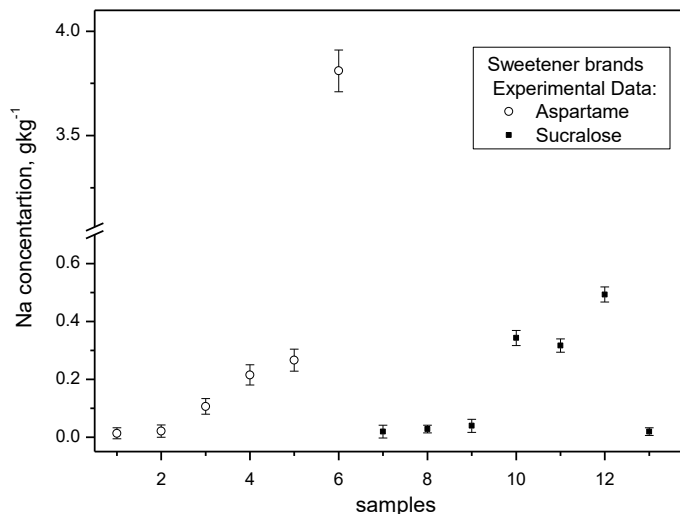
**Table 2. Na and Cl concentrations in sweetener brands**

Elements g kg <sup>-1</sup>	Sweetener Brands			
	aspartame n = 6		sucralose n = 7	
	Na	Cl	Na	Cl
MV	0.74	2.74	0.18	2.35
$\pm$ 1SD	1.51	4.50	0.20	2.74
Min	0.013	0.036	0.019	0.089
Max	3.81	11.10	0.49	6.4

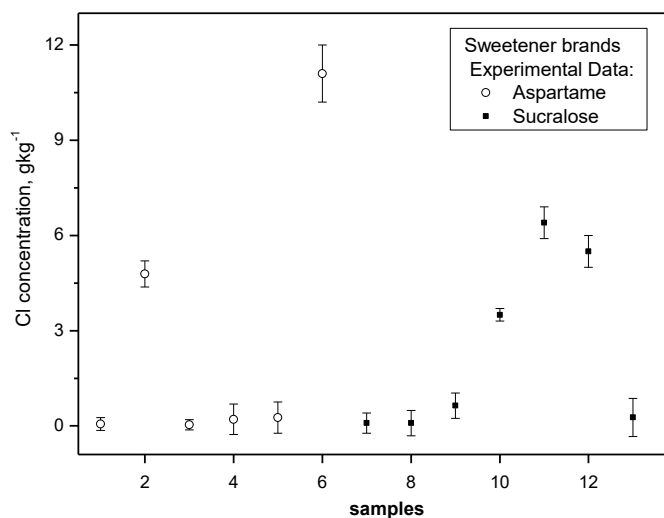
n: number of samples

According to Table 1, Z values indicated that our result were satisfactory ( $|Z \text{ score}| < 2$ ) and within the range of certified data at the 95% confidence level as well as the repeatability of the method (RSD < 10 %).

Related to Table 2, the recommended Na intake is 1.5 g / day for adults (aged 18-50 years), with smaller amounts for the elderly, due to the decrease in energy consumption. The tolerable upper limit of intake for adults is established at 2.3 g / day [24, 25]. Only one brand is above the recommended (see Figure 1). Evaluation of this element in sweeteners is important in nutritional investigations and for consumers because excessive Na consumption is one of the major risk factors, responsible for hypertension and cardiovascular diseases [13, 26]. Related to Cl, the adequate intake for adults is established at 2.3 g / day, and gradually decreases after age 50. The tolerable upper limit of ingestion for adults is 3.6 g / day [24, 25]. The present results emphasize a high concentration in many samples.



**Figure 1. Na concentration in brands of sweeteners samples**



**Figure 2. Cl concentration in brands of sweeteners samples**

### 3. CONCLUSIONS

The analyses using the NAA technique provide the knowledge of elemental composition of the sweeteners. The elements Cl and Na were identified in all samples while Ca, Fe e S were identified, only in a few samples, as trace elements. Cl and Na concentrations in the sweeteners samples showed low content for most of the brands. These values obtained do not represent a potential danger for human health.

These evaluations are important for consumers to control the consumption of sweeteners, mainly patients with Metabolic Syndrome (~ 2000 million people in Brazil), as well as to contribute with information in nutritional investigations.

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