



Analytical Methods

Determination of elemental content in solid sweeteners by slurry sampling and ICP OES

Rafael A. Sousa, Nivaldo Baccan, Solange Cadore *

Institute of Chemistry, University of Campinas, POB 6154, 13083-970 Campinas, SP, Brazil

ARTICLE INFO

Article history:

Received 14 December 2009

Received in revised form 12 July 2010

Accepted 18 July 2010

Keywords:

Elemental content
Solid sweeteners
Slurry sampling
Direct analysis
ICP OES

ABSTRACT

Inorganic species (Ca, Cd, Cu, Co, Fe, Mg, Mn, Na, Ni, Pb, Se and Zn) were determined in solid sweeteners by ICP OES without employing a mineralization step. Robust conditions were used in the ICP equipment and the samples were analyzed as aqueous suspensions. The analysis of different kinds of sweeteners showed different concentration profiles. Some samples presented contamination by Cd but, in general, the samples were found to contain Ca, Fe, Mg, Mn, Na and Se in tolerable amounts. The method accuracy was evaluated and recovery values between 90% and 110% were attained for the recovery experiments, the RSD values were lower than 5% in most cases and the method was shown to be adequate for monitoring inorganic elements in this kind of sample.

© 2010 Elsevier Ltd. All rights reserved.

1. Introduction

Edulcorants are chemical molecules that have a sweet taste and a sweetness power much superior to the sweetness of “natural sugar” (sucrose). The substitution of sucrose by edulcorants allows producing foods of lower caloric content with satisfactory sweetness (Kretchmer & Hollenbeck, 1991). Dietetic sweeteners are important examples of products formulated with natural and/or synthetic edulcorants in order to provide sweetness to foods and beverages (ANVISA, 1998). In Brazil, some of the most used edulcorants are: cyclamate, saccharine, aspartame and stevioside (Toledo & Ioshi, 1995) and they were considered in this work.

It has been observed that the amount of sugar ingested by people has been increasing and the excessive amounts of sugar are being considered the cause of many human diseases, such as dental caries and obesity.

In this scenery, the sweeteners are used by people with restrictions to the ingestion of sucrose, fructose and glucose, such as the obese and, mainly, diabetics (Mitchell, Dedman, & Garman, 2001). Thus, the use of sweeteners has been winning social and economic importance (Henin, 2001; Musto, Lim, & Suslick, 2009).

On the other hand, the sweeteners are not perfect substitutes for sugar and side effects have been observed for all the cited molecules. Historically, there were few concerns about the safe use of sweeteners but nowadays maximum daily ingestion levels are suggested by biochemists (Kretchmer & Hollenbeck, 1991) and also by the sweetener manufacturers.

Recent papers reported the presence of inorganic species in liquid dietetic sweeteners, as constituents or contaminants. For liquid cyclamate–saccharine sweeteners Cd was found in one of 15 analyzed samples, employing the ICP OES technique (Sousa, Baccan, & Cadore, 2006), As and Pb were found in two of four samples of liquid aspartame sweeteners, analyzed by ICP-MS (Sousa, Ribeiro, Vieira, Curtius, Baccan, & Cadore, 2007) and Se was found in many samples of liquid sweeteners, containing cyclamate–saccharine and stevioside, analyzed by GF AAS (Sousa, Santos, Baccan, & Cadore, 2009).

Considering these data and other applications of Inductively Coupled Plasma Optical Emission Spectrometry (ICP OES) for the determination of major and minor elements in foods (Pedro, Oliveira, & Cadore, 2006), this technique was chosen for the analysis of different solid sweeteners having cyclamate–saccharine, stevioside or aspartame contents.

The present work reports a systematic study about the determination of Ca, Cd, Co, Cu, Fe, Mg, Mn, Na, Ni, Pb, Se and Zn in solid sweeteners as aqueous suspensions (some samples do not dissolve completely in water or dilute nitric acid). For this purpose a cross-flow nebulizer was used and the analyses were carried out under robust conditions.

2. Materials and methods

2.1. Reagents, solutions and cleaning procedures

Analyte stock solutions at 1000 mg L⁻¹ in 2% v/v HNO₃ were prepared with Titrisol (Merck) standards, concentrated HNO₃ (Car-

* Corresponding author. Tel.: +55 19 3521 3125; fax: +55 19 3521 3023.
E-mail address: cadore@iqm.unicamp.br (S. Cadore).

lo Erba, PA) and deionized water (18.2 MΩ cm). From these solutions multielementar standards were prepared in 0.3% (v/v) HNO₃ to build the analytical curves for Ca, Cd, Co, Cu, Fe, Mg, Mn, Na, Ni, Pb, Se and Zn. For a mineralization procedure (used for comparison measurements) concentrated nitric acid and hydrogen peroxide (30% (m/m), Carlo Erba, PA) were used. All the laboratory materials were previously cleaned in a 10% (v/v) HNO₃ bath for at least 12 h and then washed with deionized water.

2.2. Samples (types and preparation procedure)

Commercial solid sweeteners having cyclamate–saccharine, stevioside or aspartame contents were used. Since some sweetener samples do not dissolve completely in water or dilute nitric acid, due to the presence of SiO₂ as an additive, sample suspensions were prepared by mixing 1 g of sample with ca. 25 mL of 0.3% v/v HNO₃.

For the mineralization assay 1 g of sample was mixed with 10 mL of concentrated nitric acid and 8 mL of hydrogen peroxide. This mixture was heated on a hot plate at ca. 60 °C close to dryness and then diluted to 25 mL with deionized water.

2.3. Analysis (instrument conditions and figures of merit)

The analytical measurements were made with a simultaneous Perkin–Elmer ICP OES, model Optima 3000DV, equipped with a peristaltic pump, a cross-flow nebulizer (coupled to a Ryton double pass spray chamber) and a ceramic central torch tube injector with an internal diameter of 2 mm.

The sample solutions were analyzed, immediately after manual agitation, using the instrumental conditions presented in Table 1.

Under the conditions shown in Table 1 the accuracy of the analyses were evaluated through addition and recovery experiments and also by a comparison between the results obtained for a sample analyzed without a mineralization step and after its mineralization in acidic media.

The limits of detection were calculated as suggested by Thomsen, Roberts, & Burgess (2000): LOD = (3 RSD BEC)/100. In this formula the RSD is the relative standard deviation for ten measurements of the blank solution and the BEC is the “background equivalent concentration” (calculated for the instrumental conditions used).

3. Results and discussion

The samples studied containing cyclamate–saccharine, stevioside and aspartame correspond to ordinary sweeteners used in Latin America, while aspartame is the principal sweetener used worldwide (Kretchmer & Hollenbeck, 1991). According to information found on the sample labels these sweeteners contain lactose, silica and edulcorants (cyclamate–saccharine or stevioside or aspartame).

Experimentally, it was observed that some samples did not dissolve completely in water or dilute nitric acid and this can be explained by the presence of silica. This factor was confirmed by a semi-quantitative analysis using X-ray fluorescence (microEDX, Shimadzu, model 1300).

The Si content obtained was 2% (w/w) for a test sample containing cyclamate and saccharine. Thus this kind of sample must be analyzed as an aqueous suspension or after a mineralization with hydrofluoric acid (Oliveira, 2003).

Considering sample preparation facilities and aspects related to the torch integrity the former option was selected for this work. The samples were prepared as aqueous suspensions and the dilu-

Table 1
Instrumental conditions used in the ICP OES equipment.

RF power (W)	1300
Nebulization flow rate (L min ⁻¹)	0.6
Nebulization system	Cross-flow nebulizer coupled to a double pass spray chamber
Auxiliary argon flow rate (L min ⁻¹)	0.6
Main argon flow rate (L min ⁻¹)	15
Pumping flow rate (mL min ⁻¹)	1.0
Wash flow rate (mL min ⁻¹) and time (s)	2; 15
Background correction	2 points
Integration time (s) and read delay (s)	1–5; 30
Replicates	3
Internal standard*	Y II: 371.030
Torch configuration	Axial
Elements (λ/nm)	Ca II: 317.933; Cd I: 228.802; Co II: 228.616; Cu I: 327.390; Fe II: 238.204; Mg II: 280.270; Mn II: 257.610; Na I: 330.237; Ni II: 232.000; Pb II: 220.353; Se I: 196.026; Zn I: 213.856

(I) Atomic emission line.

(II) Ionic emission line.

* The internal standard was used at 1 mg L⁻¹ for the determination of Ca, Co, Fe, Mg, Mn, Ni and Zn.

tion factor used was based on the limitations of the cross-flow nebulizer, which supports up to 5% (w/w) of dissolved solids.

In relation to the instrumental conditions, robust plasma was used to minimize possible interferences of organic matter and sodium (Brenner & Zander, 2000). The selected conditions (Table 1) were based on a previous work where the instrument optimization was systematically evaluated for liquid dietetic sweeteners (Sousa et al., 2006).

Since there is not a reference material for these kinds of sweeteners, addition and recovery experiments were used in order to evaluate the method accuracy (Thompson, Ellison, Fajgelj, Willetts, & Wood, 1999), and also a comparison between a sample analyzed directly (in acidic solution) and after an acidic mineralization. The results obtained for the accuracy evaluation are presented in the data in Table 2 shows that the average recoveries were between 90% (Pb) and 97% (Cd) for most analytes, indicating a good accuracy for these determinations. In addition, the presence of undissolved solids did not clog the nebulizer and the RSDs obtained were lower than 5%.

Table 2

Recoveries (%) obtained for the analytes added to three different solid sweeteners (containing cyclamate–saccharine, stevioside or aspartame).

Analyte	Average recovery	Recovery range	
		Low level ^a	High level ^b
Ca	96	96–103	94–97
Cd	97	94–100	89–100
Co	93	88–101	96–100
Cu	97	92–94	97–105
Fe	94	88–103	90–99
Mg	97	98–102	96–100
Mn	94	92–98	88–101
Na	83	75–78	89–91
Ni	94	90–115	92–95
Pb	90	96–106	93–103
Se	98	80–119	91–102
Zn	92	88–103	90–98

^a 0,05 mg L (Cd, Pb, Se), 0,1 mg L (Co, Cu, Fe, Mn, Ni, Zn) and 2 mg L (Na, Ca, Mg).

^b 0,10 mg L (Cd, Pb, Se), 0,2 mg L (Co, Cu, Fe, Mn, Ni, Zn) and 4 mg L (Na, Ca, Mg).

Download English Version:

<https://daneshyari.com/en/article/1185737>

Download Persian Version:

<https://daneshyari.com/article/1185737>

[Daneshyari.com](https://daneshyari.com)