



Analytical Methods

A new approach for the determination of sulphur in food samples by high-resolution continuum source flame atomic absorption spectrometer

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ABSTRACT

The new approach for the determination of sulphur in foods was developed, and the sulphur concentrations of various fresh and dried food samples determined using a high-resolution continuum source flame atomic absorption spectrometer with an air/acetylene flame. The proposed method was optimised and the validated using standard reference materials, and certified values were found to be within the 95% confidence interval. The sulphur content of foods ranged from less than the LOD to 1.5 mg g⁻¹. The method is accurate, fast, simple and sensitive.

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1. Introduction

Sulphur and sulphur compounds are found naturally in some vegetables and fruits. Also, they are widely used in food processing as preservatives in a range of foods and beverages to prevent oxidation, inhibit bacterial growth, and control enzymatic-browning (Lück & Jager, 1995; Zhong et al., 2012).

Classic and standard methods to determine sulphur, and related compounds, in foods require complicated sample pre-treatment and long preparation processes. After sample preparation, foods are analysed using titration (AOAC, 1994) or spectrometric and similar methods (Zhong et al., 2012). Generally, sulphur has been determined by spectrometry using a range of methods, such as inductively coupled plasma – atomic emission spectroscopy (ICP-AES) (Mroczek, Werner, & Schrön, 1998), inductively coupled plasma – mass spectrometer (ICP-MS) (Clough, Evans, Catterick, & Evans, 2006; Yu, Kelly, Fassett, & Vocke, 2001), ultraviolet–visible spectroscopy (UV–VIS) (Atanassov, Lima, Mesquita, Rangel, & Tóth, 2000; Burakham, Higuchi, Oshima, Grudpan, & Motomizu, 2004; Hassan, Hamza, & Mohamed, 2006; Kass & Ivaska, 2001; Kurzawa, Janowicz, & Suszka, 2001; Yang, Zhang, Korenaga, & Higuchi, 1997), X-ray fluorescence (XRF) (Necemer et al., 2003)

and chromatography (Bak, Schuhmann, & Jansen, 1993). Every method has disadvantages, such as high cost, time consumption, poor precision and selectivity, and none is free from interference (Dittrich & Vorberg, 1983; Parvinen & Lajunen, 1994; Tittarelli & Lavorato, 1987). The development of high-resolution continuum source flame and electrothermal atomic absorption spectrometry (HR-CS-FAAS) has allowed determination of sulphur in different materials (Baysal & Akman, 2011; Becker-Ross, Florek, & Heitmann, 2000; Borges, Silva, Curtius, Welz, & Heitmann, 2006; Huang, Becker-Ross, Florek, Heitmann, & Okruss, 2005, 2006; Jim, Katskov, & Tittarelli, 2011; Welz, Becker-Ross, Florek, & Heitmann, 2005; Welz, Becker-Ross, Florek, Heitmann, & Vale, 2003; Welz et al., 2009, 2010). For this purpose, diatomic molecules containing sulphur evolve in the gas phase, using a flame or graphite furnace, and the characteristic molecular absorption of sulphur measured.

In literature, there are various articles about sulphur determination using HR-CS-FAAS (Baysal & Akman, 2011; Huang et al., 2006; Jim et al., 2011; Virgilio, Raposo, Cardoso, Nobrega, & Gomes Neto, 2011). This study reports a simple, cheap and rapid method for the determination of sulphur in various foods using HR-CS-FAAS. The experimental parameters were optimised and validity of the method was tested using certified reference materials (CRMs).

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2. Materials and methods

2.1. Instrumentation

An Analytik Jena ContrAA 700 High Resolution Continuum Source Atomic Absorption Spectrometer (Analytik Jena, Jena, Germany) equipped with a 300 W xenon short-arc lamp (XBO 301, GLE, Berlin, Germany) as a continuum (radiation) source was used throughout the work. This equipment uses a compact high-resolution double echelle monochromator and a charge-coupled device (CCD) array detector with a resolution of about 5 pm per pixel. Measurements were carried out at 258.056 nm. The number of pixels in the array detector used for detecting the continuum source (CS) line(s) was five (central pixel ± 2). An oxidising air/acetylene flame was used for CS formation. All measurements were carried out as five replicates.

A Topwave microwave-assisted digestion system (Analytik Jena, Berlin, Germany) equipped with pressure controlled Teflon vessels (50 mL) were used for food digestion. Laboratory equipment was cleaned appropriately to avoid contamination.

2.2. Reagents and analytical solutions

High-purity water (resistivity 18.2 M Ω cm) obtained by a TKA reverse osmosis system (TKA Wasseraufbereitungssysteme GmbH, Niederelbert, Germany) and was used throughout the analyses. Inorganic acids and reagents of analytical grade were used (HNO₃ 65% (v/v), H₂O₂, H₂SO₄, Merck, Darmstadt, Germany). The sulphur standard was prepared by dissolving high purity sulphuric acid (Merck, Darmstadt, Germany) in water. The standard reference materials, NCS ZC 73013 spinach and NCS ZC 73016 chicken, were provided by the National Research Centre for Certified Reference Materials (Beijing, China). Foods were collected from supermarkets in Istanbul (Turkey).

2.3. Sample preparation

Food samples and certified reference materials were stored in polyethylene bags and kept at room temperature. Prior to analysis, the samples were dried at 50 °C in an air-ventilated oven for 12 h. The samples were allowed to cool over silica gel and stored in tightly closed folding polyethylene cups. All samples were ground manually in an agate mortar. Each sample weighed approximately 0.4 g and underwent microwave-assisted digestion using 7 mL of concentrated HNO₃ and a mixture of 7 mL of concentrated HNO₃ + 3 mL H₂O₂. The microwave-assisted digestion programme is shown in Table 1. Each food included different brands and each brand was analysed as 10 different samples ($N = 10$).

3. Results and discussion

3.1. Optimisation of instrumental parameters

Wavelength, flame type, air/acetylene ratio and burner height were investigated. Primary CS lines 257.593, 257.959 and

Table 1
The microwave-assisted digestion program for the digestion of food materials.

Temperature (°C)	Pressure (bar)	Ramp (sec ⁻¹)	Time (min)	Power (%)
170	50	2	5	90
190	50	5	15	90
50	50	1	10	0
50	0	1	10	0
50	0	1	1	0

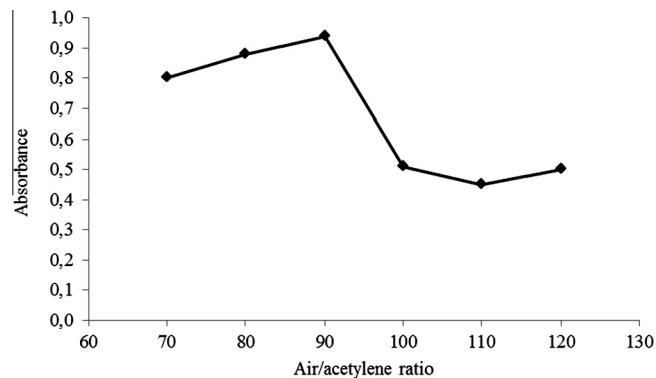


Fig. 1. Fuel rate (air/acetylene ratio) for the determination of sulphur in food material by HR-CS-FAAS.

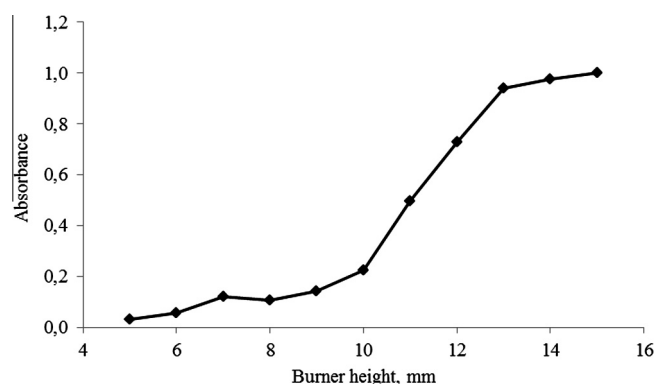


Fig. 2. Burner height for the determination of sulphur in food material by HR-CS-FAAS.

258.056 nm are well documented in the literature with respect to sensitivity and isolation. CS lines were investigated for the determination of sulphur and 258.056 nm found to be the best in terms of sensitivity and separation, and was used subsequently to determine sulphur in the samples. To optimise fuel rate (air/acetylene ratio) and burner height, digested NCS ZC 73013 spinach and 1% (v/v) H₂SO₄ was used. Rates between 60 and 120 L h⁻¹ were tested, and the maximum absorbance was achieved at 90 L h⁻¹ (Fig. 1). Similarly, as shown in Fig. 2, the maximum repeatable absorbance at 258.056 nm was obtained using a burner height of 13 mm. In the literature, both air/acetylene and nitrous oxide/acetylene flames have been used. In this study, air/acetylene was preferred for ease of use and the results obtained.

3.2. Effect of the digestion reagent for sample preparation

The two most common reagents (HNO₃ and mixture of HNO₃ + H₂O₂) were investigated for their impact on the stability, accuracy and homogeneity of digested samples. NCS ZC 73013 spinach underwent microwave digestion as shown in Table 1 and analysed immediately afterwards and, subsequently, at 3, 6 and 12 h, 1 day and 2 days. During the waiting period, all samples were kept in closed polyethylene cups in the fridge (+4 °C) and samples were equilibrated to room temperature before analysis without any recorded losses. Results for NCS ZC 73013 spinach immediately after digestion were within the 95% CI and the LOD was similar for both solutions. Sensitivity decreased using HNO₃ + H₂O₂ after three hours whereas samples were stable for almost two days using only HNO₃. Longer-term stability is important for accurate analysis and, for this reason, HNO₃ was used for digestion.

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