



Original Research Article

Assay of sodium in food: Comparison of different preparation methods and assay techniques



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ABSTRACT

Excessive salt consumption has recently awakened considerable worldwide interest because of its serious health implications. Consequently, European authorities have stated that salt content has to be clearly indicated on the food labels. In order to choose one protocol to determine the sodium content, three preparation methods (dispersion in water, wet digestion and dry ashing) and four analytical techniques (flame atomic absorption spectrometry (FAAS), flame atomic emission spectrometry (FAES), inductively coupled plasma-atomic emission spectrometry (ICP-AES) and thermometric endpoint titrimetry (TET)) have been compared. As indirect methods based on the assay of chloride could lead to wrong results, especially when food additives are involved in the recipes, it was chosen not to include them in this study. After an evaluation of analytical parameters, each combination, preparation mode and analytical technique, was tested with samples of tomato ketchup sauce. If all of them can fit to the purpose (recovery rates close to 100% and low dispersion of the results), two possibilities were pointed out: dry ashing with FAAS and the dispersion combined with TET.

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1. Introduction

Since ancient times, large amounts of cooking salt have been consumed daily worldwide for its food preservative as well as for its palatable properties. If sodium is an essential mineral for maintenance of plasma volume, acid-base balance, transmission of nerve conductance and normal cell function (WHO, 2012), an excessive amount of sodium can, on the other hand, be unbeneficial. Indeed, over the last few years, many evidences suggested an increase of cardiovascular diseases, renal diseases, strokes and osteoporosis risks due to excessive salt consumption (Penner et al., 2007; Morris et al., 2008). It has also been demonstrated that a lower daily salt consumption would induce an actual blood pressure decrease (Elliott et al., 1996; Whelton et al., 2002).

According to the World Health Organization guidelines (WHO, 2013), the maximum recommended daily dose of sodium for an

adult is 2 g, which corresponds to 5 g of salt (if NaCl). However, due to lifestyle and bad dietary habits, the population from developed countries is currently consuming excessive amounts of salt (the media mentioned a mean of 9 g a day (Test achats, 2014)). In daily food, the largest sources of sodium are processed meats, breads, sauces or biscuits (Havas et al., 2004; Capuano et al., 2013). Large amount of sodium could also be found in so-called “health food” in which salt is used to replace the flavor of the missing fat. The excessive salt intake becoming a world health issue, the European Union has adopted a regulation requiring the exact salt content to be clearly indicated on food labels (EU, 2011a, 2011b). Following this, salt concentration has to be calculated as 2.5 fold the total sodium content. Thus actual quantity of salt in food has to be quantified. According to the literature sources, this can be determined by two ways: directly, through the quantification of sodium or indirectly, by the determination of the chloride content and the conversion to sodium with the hypothesis that sodium is only present in the sodium chloride form (Capuano et al., 2013). Direct methods require the use of flame atomic absorption (FAAS) or emission (FAES) spectrophotometry (Orzaez Villanueva et al.,

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2000), inductively coupled plasma-atomic emission spectrometry (ICP-AES) (Fingerova and Koplík, 1999), ion selective electrode (AOAC, 1990) and a new method by thermometric endpoint titrimetry (TET) (Smith and Haider, 2014). The indirect chloride based approach often involves the use of titrimetry or ionic chromatography. However, as the European Union allows the use in food of 190 sodium containing additives as well as 15 different chlorine containing additives (EU, 2008, 2011a, 2011b) for specific reasons (preservatives, leavening agents, “salt replacer” like potassium chloride (Busch et al., 2013), ...), even if the major source of sodium in foods is the common salt (He and MacGregor, 2010), indirect methods could lead to incorrect values. Capuano et al. (2013) and the Codex Alimentarius (2005) have demonstrated these differences. As recent trends indicate that the indirect methods should not be used, the aim of this study was to evaluate different protocols using the direct approach (in which some of them follow an official method) in order to select objectively the best global protocol. From this perspective, twelve protocols resulting from the three common recipes for the sample preparation combined with four analytical techniques (three “traditional” based on atomic spectroscopy and the recently developed method (TET)) were compared on ketchup tomato sauces. For each protocol, accuracy and precision have been evaluated and compared to each other by using statistical tools. The entailed costs have also to be included in order to make the final choice.

2. Material and methods

2.1. Reagents

All reagents were of analytical grade. From Chem-Lab (Zeldegem, Belgium), we purchased Aluminum nitrate nonahydrate (purity $\geq 98.5\%$), ammonium hydrogen difluoride (purity $\geq 98.5\%$), Nitric acid ($\geq 65\%$ m/m) and Potassium nitrate (purity $\geq 99\%$). Ammonium hydroxide “baker grade” ($\geq 25\%$ m/m) was from J.T. Baker (Deventer, the Netherlands). Potassium chloride “max 0.005% Br” (purity $\geq 99.5\%$), Hydrochloric acid fuming ($\geq 37\%$ m/m) and Sodium chloride (purity $\geq 99.5\%$) were from Merck (Darmstadt, Germany). The latter was dried (i.e. 120°C) for at least 2 h before its use for the preparation of the standard solutions. Solutions were prepared with ultra-pure water (electrical resistivity of $18.2\text{ M}\Omega\text{ cm}$) generated by Milli-Q device (model Synergy UV; Merck Millipore; Molsheim, France). Laboratory vessels were immersed overnight in a bath of 50% nitric acid (v/v) solution, thoroughly rinsed with Milli-Q water and dried before use.

2.2. Experimental design

Three types of ketchup were analysed: a common one found in a supermarket, a second one without added sodium specific for people seeking low sodium diet and a third, home made, spiked sample. The last one was prepared with the low sodium tomato sauce in which a precise and known amount of sodium chloride was added. The homogeneity was assured by an overnight shaking of the preparation. This sample was made in order to evaluate the method accuracy. Each sample was prepared by three methods in ten replicates. We finally obtained for each kind of tomato a first batch of 10 replicates sauce after dissolution in water, a second batch of 10 replicates after acid digestion and a third batch of 10 replicates after dry ashing. The resulting solution was analysed by four analytical techniques: FAAS, FAES, ICP-AES and TET. This preparation of individual batches made of ten replicates allowed us to make a representative statistical evaluation which can lead to an objective comparison. All data were entered into a Microsoft Excel spread sheet in order to perform all the calculations and the statistical analysis.

2.3. Sample preparation

2.3.1. Dispersion in water

About 6 g of tomato sauce were precisely weighed using an analytical balance (model CP224S; Sartorius, Göttingen, Germany) and 30 mL of purified water were added. The mixture was heated to boiling under reflux with stirring. After cooling, the solution was transferred into a bottom flask and made up to 100 mL with water. The supernatant was sampled and, if necessary, diluted according to the analytical technique described below.

2.3.2. Wet digestion

About 6 g of tomato sauce were precisely weighed and digested by 5 mL of nitric acid and heating to boiling under reflux for 1 h. After cooling, mixture was transferred into a bottom flask and made up to 100 mL with water. The resulting solution was sampled and, if necessary, diluted according to the analytical technique described below.

2.3.3. Dry ashing

About 6 g of tomato sauce were precisely weighed in a porcelain crucible. Samples were first burned with Bunsen burner then heated at 550°C during at least 8 h in a muffle oven (model VMK135; Kontron Spektral Analytik, Eching, Germany) to complete the pyrolysis. After cooling, ashes were treated with 1 mL of concentrated hydrochloric acid. Covered crucible was heated on a hot plate at 120°C during 15 min. After cooling, the solution was transferred into a bottom-flask and made up to 100 mL with water. The resulting solution was sampled and, if necessary, diluted according to the analytical technique described below.

2.4. Instrumentation

2.4.1. FAAS procedure

FAAS measurements were performed using a Perkin Elmer 1100B Atomic Absorption Spectrometer (Perkin Elmer Inc., Waltham, MA, USA) with an oxidizing air-acetylene flame and hollow cathode lamp (model P971; Photron PTY. Ltd., Narre Warren, Victoria, Australia) as a radiation source. Acetylene $\geq 99.5\%$ of purity was purchased from Messer (Zwijndrecht, Belgium). Instrumental parameters were set as recommended by the manufacturer (standard condition for atomic absorption, Perkin Elmer, 1982). As it indicates only “oxidizing flame”, the flow of acetylene was optimized to maximize the absorbance signal and minimize the background set (also called the signal to background ratio, SBR). These operating conditions are shown in Table 1.

First, in FAAS measurements, the presence of a small amount of acid contribute to the stability of the solutions. Second, as ionization of the sodium due to the temperature of the flame causes depression of the signal, it has been proposed to add another metal with a high ionization potential (e.g. potassium) in sufficient quantity to overcome this effect (Caroli, 2007). According to this, standards as well as diluted sample solutions were acidified to 1% (v/v) with concentrated hydrochloride acid and spiked to 200 mg L^{-1} of potassium by addition of a potassium chloride

Table 1
Operating conditions for flame atomic absorption spectrometry (FAAS) measurements.

Fuel gas flow rate (L min^{-1})	1
Support gas flow rate (L min^{-1})	8
Burner length (cm)	10
Lamp current (mA)	12
Wavelength (nm)	589.0
Slit width (nm)	0.2
Integration time (s)	3
Number of read cycles	3

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