Detection of sulfites in fresh meat preparation commercialised at retail in Lazio Region

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Abstract

In Europe sulfur dioxide (SO_2) and sulfites in foods and beverages at concentrations of more than 10 mg kg^{-1} or 10 mg L^{-1} expressed as SO_2 equivalents are subject to mandatory labelling. In fresh meats the addition of sulfites is not admitted because of their unlawful use to give the product a more attractive appearance. Aim of the study was to detect sulfites in meat product sampled in 19 commercial shops of Lazio Region. In n=12 samples, sulfites were present at different concentrations and no indication for them was reported on the label. Sulfites concentrations ranged from 13.3 to 1278.9 mg kg^{-1}. The results of the present investigation underline the need for better controls by operators, not only under the food information but also in the consumers' health perspective.

Introduction

There is evidence that the prevalence of food allergies and intolerances has increased over the last decades (Kosunen *et al*., 2002; Marrone *et al*., 2012; Piccolo *et al*., 2016), even if their actual prevalence in developed and developing countries is still uncertain (EFSA, 2014).

In the European Union (EU), in order to prevent and manage food adverse reactions and to protect the health of vulnerable consumers, labelling of foods and ingredients with a scientifically proven allergenic or intolerance effect is established by the Regulation (EU) No 1169/2011 (European Commission, 2011). Annex II of this Regulation provides a detailed list of these substances that include sulfiting agents. Sulfites are a group of compounds composed by sulfur dioxide and several inorganic sulfite salts that may liberate SO_2 under appropriate conditions. They are usually added in a large variety of foodstuffs and beverages (fish, potatoes, wine, dried fruits, *etc*.) to prevent oxidation and bacterial growth (Taylor *et al*., 1986) and then to prolong shelf-life. Sulfites can also enhance food appearance by inhibiting discoloration (Ruiz-Capillas and Jiménez-Colmenero, 2009) due inhibiting enzymatic and Maillard-type browning reactions (Vandevijvere *et al*. 2010).

However the ingestion of sulfite-containing foods may cause allergic reaction and food intolerance symptoms in sensitive individuals (Taylor *et al*., 1986), such as asthmatic reactions and bronchospasm, occasionally severe, hives, flushing, bradycardia, as well as prominent gastrointestinal symptoms (Schwartz, 1983). Moreover, sulfites may affect the nutritional quality of food by interacting with some vitamins (such as thiamine, folic acid, pyridoxal and nicotiamide) promoting their degradation (Pizzoferrato *et al*., 1990).

An acceptable daily intake (ADI) of sulfites (expressed as SO_2) has been defined as 0.7 mg kg^{-1} body weight (FAO/WHO, 2007). In European Union, Directive 95/2/EC (European Commission, 1995) and Directive 2006/52/EC (European Commission, 2006) fix maximum levels of sulfites for several foods and beverages expressed as SO_2 equivalents in mg kg^{-1} or mg L^{-1}, which refer to the total quantity available from all sources. Moreover, Regulation (EC) No 1169/2011 (European Commission, 2011) lays down that sulfur dioxide and sulfites in foods and beverages at concentrations of more than 10 mg kg^{-1} or 10 mg L^{-1} are subjected to mandatory labelling.

In fresh meat preparations, the addition of sulfites to preserve the product appearance is prohibited. Therefore, the value of 10 mg kg^{-1} may be considered as a maximum residual limit (MRL). This latter value could be exceeded if, for example, white wine or other ingredients containing sulfites are present in the food formulation. So, a fresh meat preparation could show relatively high level of sulfites even though it has never been treated with sulfiting agents.

The aim of the present paper was the detection of sulfiting agents in some fresh meat preparations (hamburger and sausages) and to propose a maximum allowable limit for sulfiting agents taking into account that their addition is illegal but, due to use of some ingredients or because of matrix degradation, a residual amount can be found.

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Materials and Methods

Experimental tests were carried out on 38 samples of fresh minced meat preparations (pork's hamburger and sausages) collected at retail during the official controls in 19 stores of Lazio Region.

The screening-qualitative analyses were performed by using AOAC method for sulfites in meats (AOAC, 1995). In particular, 10 grams of homogenised sample were placed in a test tube with 10 mL of distilled water. Then, 2.5 mL of H_2SO_4 were added and the whole solution was heated in a water bath at 70°C. The test tube was sealed by interposing a strip of adsorbent paper, previously soaked in a solution of malachite green, between the cap and the glass. A discoloration of the paper occurring within a maximum time of 30 minutes indicated the presence of sulfiting agents at concentrations higher than 10 mg · kg^{-1} (expressed as SO_2) (LOQ).

The positive samples at the screening analysis were confirmed by an ion exchange chromatographic method (Iammarino *et al*., 2010) to obtain quantitative evaluations. According to this confirmatory test, a 4-g portion of the homogenised sample was mixed with 40 mL of a stabilising solution containing 50 mM NaOH, 10 mM fructose and 0.1 M EDTA in a horizontal shaker for 30 min. After centrifugation for 5 min at 250 ×g at room temperature, 2 mL of supernatant were filtered through Anotop 10 LC (0.2 µm, 10mm,Whatman) before being submit-

ted to chromatographic analysis.

Chromatographic separations were performed on a Dionex system (Dionex Corporation, Sunnyvale, CA, USA) composed of a GP50 quaternary gradient pump, an electrochemical detector (model ED40) set to conductivity mode, equipped with a temperature compensated conductivity cell, and a Rheodyne injection valve (model RH9125, Cotati, CA, USA) with a 25µL injection loop. A Dionex anion self-regenerating suppressor (ASRS II, 4mm) was used for the electrochemical suppression, at an operating current of 50 mA. All the separations were performed using an IonPac AS9-HC column (250mm×4mm i.d., particle size: 9 µm) eluted in gradient mode at a flow rate of 1.0 mLmin^{-1}. The mobile phase consisted of 8mM Na_2CO_3 and 2.3mM NaOH (A) and 24mM Na_2CO_3 (B). The solvent gradient program started with an isocratic step at 100%A for 15 min, a gradient step to 50% A and 50% B in 1 min, and then 4 min at this eluent concentration. Finally, the system was re-equilibrated for 20 min at 100% A. The plastic reservoir bottles (DX500 2 L bottles, Dionex) were closed and pressurised with pure nitrogen to 0.8 MPa. The system was interfaced, via proprietary network chromatographic software (PeakNetTM), to a personal computer for instrumentation control, data acquisition and processing (Dionex).

Results and Discussion

Results are reported in Tables 1 and 2. The samples positive to the screening test ([SO_2] > 10.0 mg kg^{-1} (LOQ)) were 12 (31.6%) (6 hamburgers and 6 sausages). These samples were collected from 11 of the 19 total shops. The quantitative determinations of sulfite content in these *suspected positive* samples were carried out by ion chromatography. These analytical determinations revealed sulfite concentrations ranging from 35 to 1278.9 mg kg^{-1}. Each result was the mean of n. 3 replications.

On the basis of the obtained results, samples were divided into two groups: a *group A* of 7 samples (4 hamburgers and 3 sausages) with sulfite concentrations lower than 35 mg kg^{-1} and a *group B* of 5 samples (2 hamburger and 3 sausages) with sulfite concentrations higher than 200 mg kg^{-1} (Table 2).

The high sulfite concentrations (>200 mg kg^{-1}) found in the samples belonging to *group B* can be attributable to illegal sulfiting agent addition. On the contrary, fraudulent use of sulfiting agents for samples belonging to *group A* seems unlikely. In fact, at this concentrations (<35 mg kg^{-1}), sulfites would not play an effective antioxidant, antimicrobial or blanching activity (Iammarino *et al*., 2012). Therefore, the presence of such residues is, in this case, more probably due to a *carry-over* effect or it could be the result of the addition of some particular ingredients containing sulfites (*e.g*. white wine). As demonstrated by some authors (Iammarino *et al*., 2012), the presence of low sulfite concentrations in fresh meat preparations could also be attributable to the reduction, by sulfate-reducing bacteria, of free sulfates naturally present in fresh meat (Jay *et al*., 2005).

Table 1. Results obtained on the analysed fresh meat preparations.

Commercial retailer	Samples	Mean±SD (mg kg^{-1})
1	Hamburger	nd
	Sausages	nd
2	Hamburger	487±6.45
	Sausages	367±6.26
3	Hamburger	1278.9±15.66
	Sausages	nd
4	Hamburger	nd
	Sausages	231.3±3.66
5	Hamburger	nd
	Sausages	nd
6	Hamburger with red chicory	14.7±1.05
	Hamburger	nd
	Sausages	nd
7	Hamburger	19.3±1.68
	Sausages	nd
8	Hamburger	28.1±2.65
	Sausages	nd
9	Hamburger	nd
	Sausages	nd
10	Hamburger	nd
	Sausages	255.7±3.68
11	Hamburger	nd
	Sausages	34.1±2.59
12	Hamburger	nd
	Sausages	34.1±3.09
13	Hamburger	nd
	Sausages	nd
14	Hamburger	nd
	Sausages	19.5±2.33
15	Sausages	nd
16	Hamburger	13.3±3.24
	Sausages	nd
17	Hamburger	nd
	Sausages	nd
18	Hamburger	nd
	Sausages	nd
19	Hamburger	nd
	Sausages	nd

SD, standard deviation; nd, not detected.

Table 2. Results obtained on the analysed fresh meat preparations and grouped according to the concentration.

Samples	Screened samples	Group A (10.0 < [SO_2] < 35mg kg^{-1})	Group B ([SO_2] >200 mg kg^{-1})
Hamburger	19	4	2
Sausages	19	3	3
Total	38	7	5

Conclusions

From a survey of 38 fresh meat preparations, quantifiable sulfite concentrations were found in 12 samples. In 7 of these samples (18% of total samples), sulfite concentrations were lower than 35 mg kg^{-1}, whereas the remaining 5 samples (13% of total samples) showed sulfite concentrations ranging from 200 to 1278.9 mg kg^{-1}, probably as a consequence of fraudulent sulfiting agents addition. The high percentage of samples with sulfite levels higher than 200 mg kg^{-1} suggests that adulteration of fresh meat preparations by means of sulfite addition is still a relatively common practice and, considering the adverse effects of these additives, more effective control measures are needed. On the basis of the distribution of sulfite concentrations found in the positive samples and taking into account the results of previous studies (Iammarino *et al*., 2012), we suggest 40 mg kg^{-1} (expressed as SO_2) as the maximum allowable limit of sulfites in fresh meat preparations above which the use should be considered illegal. In any case, the mandatory labelling for sulfite concentrations higher than 10 mg kg^{-1} (as established by the Regulation (EC) N. 1169/2011; European Commission, 2011) would be still valid.

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