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A decade of sulfite control in Serbian meat industry and the effect of HACCP

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Abstract
In total 7,351 meat preparations and fresh processed meat products were analyzed from 555 different Serbian meat producers over a 10 year period, 4½ years before and 5½ years after mandatory HACCP implementation. From the obtained results, it could be concluded that HACCP has contributed to a better alignment of practices with the legal provisions. The share of non-compliant samples dropped from 18.6% before HACCP to 8.3% after its mandatory implementation. Average sulfite concentrations for all categories of meat preparations and fresh processed meat products decreased by 43%, declining from 33.6 mg kg-1 to 19.3 mg kg-1. Typical misuse and frequent abuse of sulfites was independent of a season. Application of HACCP principles in the Serbian meat industry raised awareness about the misuse of sulfites and contributed to a better control, minimizing exposure to sulfites.

Keywords: *Sulfite; Meat preparations, Meat products; HACCP; Serbia.*

Introduction

Sulfur dioxide and sulfites comprise the group of compounds known collectively as sulfites. The major purpose of sulfites in raw meat products is credited to their antimicrobial activity (Ruiter and Scherpenisse 2011), which allows prolongation of the shelf life of these products kept in cooling environment. Even a small amount of sulfite in meat imparts a bright red color due to its antioxidant activity. Sulfites are used because they improve food appearance by controlling enzymatic and non-enzymatic browning reactions occurring from the Maillard-type reactions (Ruiz-Capillas and Jiménez-Colmenero 2009).

The concentration in different foods is expressed as sulfur dioxide (SO_2) in mg kg-1 or mg/l depending on the nature of the food and is related to the total quantity from all sources. The European legislation approves the addition of sulfites to burger meat with a minimum content of cereals and/or vegetables of 4%, breakfast sausages, and two types of traditional Spanish raw sausages, at maximum levels of 450 mg of total SO_2/kg. The same legislation also declares that a

SO_2 content of no more than 10 mg kg-1 or 10 mg/l is considered not to be present (European Commission, 2011). However, in other countries sulfur dioxide is prohibited in these kinds of foods. Thus, in the United States, foods recognized as a source of vitamin B1 (red meat) must be free of sulfites.

Sulfiting agents have also been included in the allergens list (European Commission, 2003) making it mandatory to indicate on the label of any food containing concentrations higher than 10 mg kg-1 (expressed as SO_2), for the benefit of people with a food intolerance for sulfite (European Commission, 2000). An acceptable daily intake (ADI) for sulfite is 0.7 mg kg-1 of body weight and it is considered especially important that SO_2 must be minimized in food with high thiamine content, such as red meat (FAO/WHO, 1986).

In the Republic of Serbia, Hazard Analysis and Critical Control Points (HACCP) was firstly introduced by virtue of the Veterinary Law (Serbia, 2005). Its mandatory implementation, for all animal source food producers, became effective only in June 1st of 2011 after the adoption of a new Food Safety Law (Serbia, 2009). By the end of 2012, almost all Serbian meat producers (93.5%) had an operational and certified HACCP system in place (Tomašević et al. 2013).

Throughout the western Balkan region, ground meat is raised to a form of art. Pljeskavica (plyess-ka-vee-tsa) is made from ground meat and minced onion, pounded thin, shaped like a burger and then grilled on both sides to a smoky brown product. The word "pljeskavica" comes from "pljesak", a regional word for clapping the hands, the motion used to press the burger into a thin round. With "ćevapi" (chay-vop-ee) the meat is shaped into cylinders to make it hold together well. They are formed by hand or extruded through a funnel. Both meat products are grilled over very high heat to lightly char the outside on all sides to a smoky brown and keep the inside juicy.

European and current Serbian legislation do not approve the addition of sulfites to "pljeskavica" or "ćevapi". In recognition of the significance of minced meat in the Serbian diet, the Directorate for Veterinary Affairs has never approved the addition of sulfur dioxide to minced meat sold in Serbia. Therefore, any addition of sulfites to meat preparations and fresh processed meat products (except for burger meat with a minimum content of cereals and/or vegetables of 4%) in Serbia is deemed illegal.

This survey was conducted to assess meat preparations and fresh processed meat products for SO_2 content by sampling meat producers of all sizes in geographically diverse areas of the Republic of Serbia. It was intended to obtain a representative sample of meat preparations and fresh processed meat products and to determine if changes in SO_2 concentrations had occurred in the last decade. The main objective of this study was to establish if the mandatory HACCP implementation had had an impact on misuse and control of sulfites in the Serbian meat industry. Another objective was to identify the type of meat product contributing most to the total intake of sulfites. The acquired data will also be useful for future assessments of dietary exposure of sulfites for Serbia consumers.

Materials and Methods

Meat product samples

All samples (7,351) were procured from retailers by the Center for Food Analysis (CFA), Belgrade, Serbia, and were manufactured by 555 different meat producers located throughout the country. After collection the samples were stored at 4°C and transported by road in dry ice packs to the CFA laboratory for analysis. All samples were analyzed within 3 days of procurement. Meat producers involved were of all sizes, with an estimated 70% share of the Serbian meat preparations and fresh processed meat products market. Ten years (2007 to 2016) were encompassed by this investigation, divided into two periods: the first was from January 1st 2007 until May 31st 2011 (2,929 samples from a period of 4 ½ years before mandatory HACCP implementation) and the second from June 1st 2011 until December 31st 2016 (4,422 samples from a period of 5 ½ years after HACCP became mandatory). All samples, with an average size of 400 g, were homogenized and analyzed using the enzymatic method according to the ISO procedure for determination of sulfite in foodstuffs (ISO 1988-2:1998).

Statistical analysis

All results represented the mean value of 3 replicates for each sample. Data were analyzed using SPSS Statistics 17.0 (Chicago, Illinois, USA) data analysis software. Factorial analysis of variance (ANOVA) and post hoc Tuckey analysis of Honestly Significant Difference (HSD) were used. The level of statistical significance was set at 0.05.

Results and Discussion

The analytical method was validated by determining the limit of detection (LOD), the limit of quantification (LOQ), the relative standard deviation calculated under repeatability conditions (RSD_r) and the relative standard deviation calculated under reproducibility conditions (RSD_R), according to the European procedure for screening methods (European Commission, 2002). LOD was calculated by the equation $LOD = X_{blank} + 3x\ SD_{blank}$ and $LOQ = X_{blank} + 10x\ SD_{blank}$; where X_{blank} is the mean concentration of the blank and SD_{blank} is the standard deviation of the blank. The LOD was 3.8 mg kg-1; the LOQ was 9.0 mg kg-1; RSD_r was 4.9%, RSD_R was 3.7% and the mean recovery was 96%. The analytical laboratory participated in the FAPAS© (Food Analysis Performance Assessment Scheme) proficiency testing program, with the latest sulphur dioxide in meat sample test conducted in 2016 (asigned value 491 mg kg^{-1}; range for $|z|<2$ was 430 – 553 mg kg^{-1}, recording a *z*-score of 1.9, indicating satisfactory analytical performance; FAPAS 2016).

According to this study the illegal addition of sulfur dioxide to meat preparations and fresh processed meat products in Serbian meat industry is a fact, at least in the last ten years. In the period 2007 to 2016, of 7,351 samples 911 (12.4%) contained sulfur dioxide. The number of non-compliant samples ranged from 23.3% in 2007 to a 7% in 2016 (Table 1). Over a decade the most affected were ćevapi (15.7%) and minced pork (14.7%), followed by pljeskavica (10.4%), minced beef (10.1%) and fresh sausages (8.9%). The seriousness of the situation is perhaps best described by the following example. In the 2010 survey, 193 samples of ćevapi were analyzed. Of these, 60 samples (31%) contained sulfur dioxide (Table 1). However, the big share of positive samples discovered in this study is still smaller then percentage for presence of sulfites reported for Spain. There it was previously reported that 65.38% of beef and pork burgers (Zubeldia Lauzurica and Gomar Fayos 1997), 62.5% of the uncooked burgers (Armentia

Alvarez et al. 1993) and 62% of the sausages analyzed (Paíno-Campa et al. 1991) contained levels of total SO_2 above 450 mg kg-1. Most recent results reported for Spain state that 18% of meat samples exceeded the maximum level of total sulfite (Pena-Egido et al. 2005), very similar to the results observed in this study in a period before mandatory HACCP implementation where 18.6% of the samples were found to be non-compliant (Table 2).

The presented investigations also revealed that in the last 10 years the highest average level of sulfur dioxide 43.6±83.8 mg kg-1 (n=458) was observed in 2007, which is not significantly different from the ones observed in 2009 and 2010 (Table 1). It was considered highly unlikely that the addition of sulfur dioxide is due to lack of knowledge within the meat industry. It must be concluded that by adopting this practice, a significant number of Serbian meat traders deliberately violates the law and risk prosecution before mandatory implementation of HACCP.

A recent study of the Serbian meat industry reported that 93.5% of the producers surveyed, claimed they had a fully operational and certified HACCP system in place, whilst the remaining 6.5% had a HACCP system in place which was not certified (Tomašević et al. 2013). Strong affirmative effect of mandatory HACCP implementation on process hygiene indicators in Serbian meat facilities was already confirmed (Tomasevic et al. 2016). However, the smaller enterprises are reported to experience problems in introducing such HACCP based systems, due to factors such as awareness of the legislation and requirements, cost of implementation, availability of systems and training (Djekic et al. 2011, Tomašević et al. 2013).

Current research revealed that average sulfur dioxide concentrations in all investigated categories of Serbian meat products reduced by 43% since meat producers report the levels of additives used and provide documentation with the amounts and concentrations of additives purchased/used in a specified production period, as obligatory due to the implemented HACCP legislation. The average sulfite concentrations significantly decreased from 33.6±73.8 mg kg-1 (n=2,929) before, to 19.3±63.1 mg kg-1 (n=4,422) after the period of mandatory HACCP implementation. It was also discovered that mandatory HACCP implementation has contributed to a better alignment of practices with the legal provisions, since the percentage of non-compliant samples dropped from 18.6% (15-870 mg kg-1) to 8.3% (11-898 mg kg-1) (Table 2). The current situation in Serbia is obviously quite similar to the one observed in Italy during the last 3 years, where 6.4% of meat product samples were positive at a sulfur dioxide screening test and 2.8% of these samples showed sulfite concentrations in the range 67.6–1437 mg kg-1 (Iammarino et al. 2017). Slightly lower data were reported for Hong Kong where 4.7% of the meat product samples contained sulfur dioxide at levels between 23 and 3,300 mg kg-1 (Chan, 2007).

The minimum share of non-compliant samples after the mandatory HACCP implementation was observed in minced beef (2.5%), followed by fresh sausages (5.4%), pljeskavica (5.9%) and ćevapi (10.6%). The least positively affected group of meat products investigated was minced pork although the average sulfite concentrations significantly decreased from 30.2±69.7 mg kg-1 (n=344) to 22.8±58.9 mg kg-1 (n=315). The share of non-compliant minced pork samples dropped only by 1.3% with the introduction of mandatory HACCP implementation (Table 2). The use of sulfur dioxide in minced pork continues to be an issue in Serbia, contrary to the situation reported for example in Scotland. There, only 2% of samples contained this

preservative in a period of 2000-2002 (Mackie 2005) while no Scottish chain retailers sold minced pork containing sulfur dioxide in recent years (Bradley 2013).

Of 1,689 samples analyzed in this study for sulfites in summer, 14.1% were assessed as non-compliant. Almost the same condition was observed in winter where 13.8% of 2,033 samples were found positive for the presence of sulfur dioxide (Table 3). This it seems that typical misuse and frequent abuse of sulfites in Serbian meat industry is independent of the season. Average sulfite concentrations observed in spring 21.5±59.4 mg kg-1 (n=2,288), summer 25.8±68.4 mg kg-1 (n=1,689) and autumn 24.9±74.2 mg kg-1 were not found to be significantly different (Table 3).

Despite the obviously positive influence of the introduction of HACCP in Serbian meat industry to control use of sulfites, the actual situation on the Serbian market is still far away from the conclusion that sulfites should be given the lowest weight as chemical hazards as it was the case in Belgium (Baert et al., 2011). For the Belgian market this conclusion could only be drawn after a study of Vandevijvere et al. (2010), who showed that sulfites intake was below the ADI in Belgium. To achieve this optimal situation in Serbia veterinary inspectors should increase the frequency of official controls for the presence of sulfur dioxide in meat and meat products, identify ways of eliminating illegal practices and prosecute violators of Food Law regulations, as to stop misuse and potential unhealthy effects of this practice for consumers.

Conclusions

Although it is obvious that HACCP raised awareness among Serbian meat producers about the misuse and control of sulfites, the results of this study show that the actual situation on the Serbian market is far from ideal. Therefore the Ministry of Agriculture and the Serbian Directorate for Veterinary Affairs can find enough reasons to engage with the meat industry to highlight this continuing problem. As long as this situation does not improve, authorities have enough reason to continue to enforce food safety regulations and prosecute businesses that illegally add sulfites to meat preparations and fresh processed meat products.

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Table 1 Sulfite content (mg kg-1) in different categories of meat products over a decade.

		Minced Pork	Minced Beef	Ćevapi (Kebab)	Pljeskavica (Burger)	Fresh Sausage	Total
2007	Number of samples	33	32	163	61	169	458
	Non-compliant (%)	32.2	25	26.3	19.6	20.11	23.3
	Range non-compliant	87-338	38-265	31-373	70-303	20-353	20-373
	Mean ± SD	58.7±97.6	42.2±81.2	47.2±84.3	41.5±86.0	38.2±80.5	43.6^{a} ±83.8
2008	Number of samples	68	92	372	143	382	1057
	Non-compliant (%)	22	14.1	25.8	23.8	11.5	19.1
	Range non-compliant	66-321	66-243	39-368	40-453	41-870	39-870
	Mean ± SD	41.6±82.6	24.7±58.9	36.9±72.7	35.0±73.3	22.5±71.1	$30.7^{b,c,d}$ ±72.0
2009	Number of samples	92	68	150	52	121	483
	Non-compliant (%)	16.3	8.8	27.3	34.6	14.9	20.3
	Range non-compliant	62-236	45-418	57-277	79-279	64-255	45-418
	Mean ± SD	29.1±65.4	18.2±64.9	53.3±86.7	64.5±92.1	24.0±55.7	$37.6^{a,b}$ ±75.2
2010	Number of samples	106	83	193	73	177	632
	Non-compliant (%)	11.3	9.6	31.0	19.2	12.4	18.3
	Range non-compliant	80-266	94-280	75-341	77-319	15-272	15- 41
	Mean ± SD	23.3±61.4	20.9±59.7	59.6±90.6	39.9±82.8	22.6±58.1	$35.8^{a,b,c}$ ±74.8
2011	Number of samples	62	41	204	68	152	527
	Non-compliant (%)	3.2	4.9	9.3	11.7	7.2	7.9
	Range non-compliant	161-199	194-211	11-316	14-274	66-249	14-316
	Mean ± SD	8.4±31.8	12.4±43.6	19.8±58.9	18.3±52.5	15.33±47.6	16.42^{e} ±51.2
2012	Number of samples	28	8	319	97	205	657
	Non-compliant (%)	10.7	0	13.5	6.2	9.8	10.9
	Range non-compliant	27-193	0	11-324	12-241	62-260	11-324
	Mean ± SD	15.3±43.7	3.9±3.53	27.82±69.9	11.8±40.4	18.8±51.9	$21.8^{d,e}$ ±59.7
2013	Number of samples	56	12	341	131	212	752
	Non-compliant (%)	7.1	0	10.6	6.9	3.8	7.6
	Range non-compliant	105-247	0	17-391	135-298	81-332	17-391
	Mean ± SD	16.2±51.4	3.52±2.88	25.3±71.7	16.3±53.1	10.7±43.4	18.6^{e} ±59.9
2014	Number of samples	86	38	537	202	227	1090
	Non-compliant (%)	8.1	5.3	11.2	5.4	6.2	8.6
	Range non-compliant	104-241	30-120	48-501	92-344	112-359	30-501
	Mean ± SD	16.7±49.1	6.5±19.4	26.9±74.0	12.5±45.5	16.8±58.3	20.6^{e} ±63.5
2015	Number of samples	99	16	334	382	296	1127
	Non-compliant (%)	24.2	0	6.8	6.3	4.4	7.5
	Range non-compliant	49-217	0	52-605	45-381	51-572	45-605
	Mean ± SD	29.6±51.4	3.9±3.4	17.3±63.0	14.9±53.0	10.0±44.8	15.5^{e} ±53.9
2016	Number of samples	29	7	210	168	154	568
	Non-compliant (%)	17.3	0	10.9	4.8	2.6	7.0
	Range non-compliant	76-479	0	82-549	140-463	189-898	76-898
	Mean ± SD	49.2±118.5	4.1±3.8	35.6±103.4	17.1±68.7	12.3±76.7	$24.1^{c,d,e}$ ±88.1
Total	Number of samples	659	397	2,823	1,377	2,095	7,351
	Non-compliant (%)	14.7	10.1	15.7	10.4	8.9	12.4
	Range non-compliant	27-479	30-418	11-605	12-463	15-898	11-898
	Mean ± SD	$26.6^{A,B}$ ±64.8	18.9^{B} ±55.6	31.9^{A} ±76.9	21.4^{B} ±62.3	18.7^{B} ±60.4	$25.0^{A,c,d,e}$ ±67.9

Means not sharing same capital letter in the same row are different significantly at the 0.05 level; Means not sharing same small letter in the same column are different significantly at the 0.05 level.

Table 2 Sulfite content (mg kg-1) in different categories of meat products before and after mandatory HACCP implementation.

	Before mandatory HACCP				After mandatory HACCP			
Group of samples	Number of samples	Non-compliant (%)	Mean ± SD	Range of non-compliant samples	Number of samples	Non-compliant (%)	Mean ± SD	Range of non-compliant samples
Minced pork	344	15.7	30.2^{A}±69.7	49-321	315	14.4	22.8^{B}±58.9	27-479
Minced beef	307	11.7	22.4^{A} ±61.3	45-418	90	2.5	7.1^{B} ±25.21	29-120
Ćevap (Kebab)	981	25.6	44.6^{A} ±81.2	39-605	1,842	10.6	25.2^{B} ±73.8	11-605
Pljeskavica (Burger)	364	21.7	38.5^{A} ±78.2	40-453	1,013	5.9	15.3^{B} ±54.2	13-463
Fresh Sausage	933	13.4	25.1^{A} ±67.4	15-870	1,162	5.4	13.5^{B} ±53.7	51-898
Total	2,929	18.6	33.6^{A} ±73.8	15-870	4,422	8.3	19.3^{B} ±63.1	11-898

Means not sharing same capital letter in the same row are different significantly at the 0.05 level.

Table 3 Seasonal variation of sulfite content (mg kg-1) in meat products.

Season	Number of samples	Non-compliant (%)	Mean ± SD
Spring	2288	10.9	21.5^{A}±59.4
Summer	1689	14.1	$25.8^{A,B}$ ±68.4
Autumn	1341	10.7	$24.9^{A,B}$ ±74.2
Winter	2033	13.8	30.0^{B} ±76.1

Means not sharing same capital letter in the same column are different significantly at the 0.05 level.