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Original research paper

# POLYCYCLIC AROMATIC HYDROCARBONS AS LIMITING PARAMETERS IN TRADITIONAL PRODUCTION OF DRY-CURED MEAT PRODUCTS

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Abstract: The goal of the study was to investigate the concentration of 16 US EPA polycyclic aromatic hydrocarbons (PAHs) and check the sensory properties in 11 dry-cured meat products, smoked in three different ways (atmospheric simulation chamber (ATMOS), industrial chamber and traditional craft smokehouse) in one meat industry facility in Serbia. Controlled industrial production cannot provide all distinguishable and specific sensory properties of traditional drycured products, so more and more frequently meat industries decide to include the products smoked in traditional craft smokehouses in their product assortment. 16 US EPA PAHs were determined using a gas chromatography-mass spectrometry method (GC-MS). The obtained results are part of the production technology validating process of the tested smoked meat products. Total concentrations of benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluranthene and chrysene (comprising the so-called "PAH4" group) in traditional homemade smoked bacon produced in compliance with all the guidelines of good manufacturing and hygiene practice, reached 15.82 µg/kg what was above the legal limits set by Serbian legislation. This indicates that there is a need for monitoring the PAH content compounds in traditional smoked meat products, and quite likely the revision of the current rulebook on traditional dry-cured meat products following the EU practice.

Key words: PAH, meat products, traditional production, ATMOS

### **INTRODUCTION**

Meat and meat products are an important part of the diet of a large number of people worldwide. The share of meat in the human diet has been constantly increasing since 1960 (Halagarda & Wójciak, 2022). In 2021, the consumption of meat per capita at the world level was 42.84 kg, while in 1961 it was 22.93 kg (FAO, 2023). In Serbia, meat consumption per capita for 2021 was 77.62 kg, of which the most consumed was pork (47.58 kg), poultry meat (16.69 kg), beef (9.15 kg), sheep and goat (4.18 kg) and other meats (0.02 kg) (FAO, 2023). Serbian traditional dry-cured meat products are famous for their geographical origin, made to recipes that are a few decades or hundreds of years old, and smoked in craft smokehouses. Consumers particularly appreciate smoked meat products made traditionally (Babić et al., 2017). Traditional meat products are generally regarded as healthy.

Research conducted by different groups of authors indicates that traditional meat products, despite their widespread acceptance by consumers, may also be associated with some health and safety issues, such as pathogens. They are often a result of large amounts of salt used during production, the presence of nitrate and nitrite residues, N-nitrosamine, biogenic amines and heavy metals, and the compounds originating from smoke i.e., polycyclic aromatic hydrocarbons (PAH) (Halagarda & Wójciak, 2022).

The list of priority PAHs varies in different countries. US EPA has listed 16 priority PAHs based on those most abundant. The EU list of 15+1 PAH is based on those considered most toxic in foodstuff. Due to its toxic potency the sum of benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluranthene and chrysene (PAH4) are of the highest concern in food (Rozentale, Zacs, Perkons, & Barktevisc, 2017). The most often extraction technique that is used for the preparation of smoked meat for PAHs determination is the Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) preparation method. In case when regulation requests a low maximum residue level of PAHs in food, it is important to develop a sensitive and reliable method with a high confidence level in results. During the year many authors used different methods for PAHs determination. The most used are gas and liquid chromatography coupled with mass chromatography, which ensured reliable results, for all 16 US EPA PAHs (Kartalović et al., 2022; Agus et al., 2023).

Controlled industrial production cannot achieve the distinguishable and specific sensory quality of traditional smoked products, so meat industries increasingly decide to have traditional craft smokehouses in their production facilities, which would allow them to enrich their product assortment with traditional smoked products made in craft smokehouses. Despite the application of HACCP, GHP and GMP, PAH compound content in the products made in this way is often above the values permitted by national regulations, with benzo(a)pyrene amounting to 2  $\mu$ g/kg, and 12  $\mu$ g/kg for the sum of PAH4 (benzo(a)anthracene, chrysene, benzo(b)fluoranthene, and benzo(a)pyrene) (Rulebook on maximal limits of contaminant residues in food, 2019). This work aims to check the content of PAH compounds in meat products smoked by different methods, to see whether PAHs are limiting factors in traditionally smoked meat production.

# MATERIALS AND METHODS

To validate the production process of smoked dry-cured meat products, 11 smoked meat products were sampled in one meat industry facility by including three different ways of smoking: ATMOS, chamber and traditional craft smokehouse. The list of samples, their shape, the smoking method, type and temperatures are listed in Table 1.

Sensory tests were performed using the accredited method sensory review of foodstuffs qualitative descriptive test, while the criteria for sensory properties were taken from the current Regulation on the quality of ground meat, meat preparations and meat products (2019).

All chemicals and reagents were HPLC grade. Anhydrous sodium acetate (NaOAc), anhydrous magnesium sulphate, CH<sub>3</sub>COONa, primary and secondary amine and C18 were purchased from "Merck" (Darmstadt, Germany), only acetonitrile (ACN) was purchased from "Sigma-Aldrich" (St. Louis, MO, USA). Sample preparation of PAHs was based on the extraction with ACN, in the presence of anhydrous magnesium sulfate and anhydrous sodium acetate, using multi-residue preparations that ensure QuEChERS, as adapted from the Association of Analytical Communities (AOAC), official method 2007.01 for extraction and clean-up, described and adjusted by Babić et al. (2018). Three grams of the sample were transferred into a centrifuge tube, with 3 mL of water and 3 mL of ACN. After intensive stirring on a vortex for 1 min, 3 g of anhydrous magnesium sulphate and 1 g of anhydrous sodium acetate were added. The sample was then centrifuged for 5 min at 3000 rpm. 1 mL of the upper (ACN) layer of the extract was transferred into the 5 mL tube, which contained 150 mg of anhydrous magnesium sulfate, 100 mg of PSA and 50 mg of C18. The tube content was centrifuged again for 5 min at 3000 rpm, obtaining a clear and pure extract.

Then, 0.5 mL of the extract was evaporated under nitrogen gas and reconstituted with hexane, resulting in a sample ready for GC-MS analysis. Analysis and calibration, to eliminate the influence of the matrix, were done according to Mastanjević, Kartalović, Vranešević, Novakov & Habschied (2020) on GC-MS (Agilent 7890B/5977A, USA). GC operating conditions were: a fused silica column (30 m x 0.25  $\mu$ m x 0.25 mm film of HP (Hewlett Packard; and injection temperature of 280 °C, using the splitless method of injection (4  $\mu$ L). The column temperature program was: 50 °C for 0.4 min; 50–195 °C at 25 °C/min, hold for

Table 1.

Product type, shape, method and smoking temperature of the samples

| No  | Meat product type                 | Shape                    | Smoking method   | Smoking type and temperature           |
|-----|-----------------------------------|--------------------------|------------------|--|
| 1.  | Carniolan sausage                 | Chunk                    | ATMOS            | Hot smoking<br>(65 °C)                 |
| 2.  | Homemade sausage                  | Chunk                    | Chamber          | Cold smoking<br>(25 °C)                |
| 3.  | Dry pork loin                     | Chunk                    | Craft smokehouse | Cold smoking<br>(25 °C)                |
| 4.  | Smoked pork loin with added water | Chunk                    | ATMOS            | Warm and hot smoking<br>(60 and 65 °C) |
| 5.  | Dry cured homemade bacon          | Chunk                    | Craft smokehouse | Cold smoking<br>(25 °C)                |
| 6.  | Kulen                             | Chunk                    | Chamber          | Warm and hot smoking<br>(25 °C)        |
| 7.  | Smoked pork loin with added water | Sliced and packed in MAP | ATMOS            | Warm and hot smoking<br>(60 and 65 °C) |
| 8.  | Budim sausage                     | Sliced and packed in MAP | Chamber          | Cold smoking<br>(25 °C)                |
| 9.  | Kamendin pancetta                 | Sliced and packed in MAP | Chamber          | Cold smoking<br>(25 °C)                |
| 10. | Smoked pork neck with added water | Sliced and packed in MAP | ATMOS            | Warm and hot smoking<br>(60 and 65 °C) |
| 11. | Kulen                             | Sliced and packed in MAP | Chamber          | Cold smoking<br>(25 °C)                |

#### Table 2.

The average values for precision, reproducibility, recovery, linearity, MQL and MDL for 16 US EPA PAHs method validation (*Source:* Adopted from Kartalović et al. (2022), copyright 2022, CC BY 4.0)

| РАНѕ                         | Precision<br>(%) | Repro-<br>ducibility<br>(%) | Recovery<br>(%) | Linearity<br>(r <sup>2</sup> ) | MQL<br>(µg/kg) | MDL<br>(µg/kg) |
|------------------------------|------------------|-----------------------------|-----------------|--------------------------------|----------------|----------------|
| naphthalene (Nap)            | 11.3             | 6.33                        | 95.0            | 0.99                           | 1.20           | 0.30           |
| acenaphthylene (Acy)         | 7.91             | 7.82                        | 99.0            | 0.99                           | 1.30           | 0.29           |
| acenaphthene (Ace)           | 8.52             | 8.32                        | 99.3            | 0.99                           | 1.05           | 0.32           |
| fluorene (Fle)               | 2.82             | 10.2                        | 100             | 0.99                           | 1.11           | 0.30           |
| anthracene (An)              | 3.53             | 3.73                        | 98.7            | 0.99                           | 1.10           | 0.30           |
| phenanthrene (Ph)            | 4.31             | 11.4                        | 85.9            | 0.99                           | 1.18           | 0.35           |
| fluoranthene (Fla)           | 3.61             | 3.72                        | 95.3            | 0.99                           | 1.15           | 0.30           |
| benz(a)anthracene (BaA)      | 9.44             | 8.6                         | 89.7            | 0.99                           | 1.30           | 0.37           |
| pyrene (Pyr)                 | 4.74             | 6.91                        | 91.1            | 0.99                           | 1.21           | 0.32           |
| chrysene (Chr)               | 5.33             | 8.20                        | 92.5            | 0.99                           | 1.13           | 0.34           |
| benzo(b)fluoranthene (BbF)   | 8.52             | 14.3                        | 86.4            | 0.99                           | 1.30           | 0.36           |
| benzo(k)fluoranthene (BkF)   | 3.51             | 3.32                        | 94.3            | 0.99                           | 1.21           | 0.32           |
| benzo(a)pyrene (BaP)         | 3.23             | 3.81                        | 96.8            | 0.99                           | 2.00           | 0.53           |
| dibenz(a,h)anthracene (DahA) | 8.72             | 11.3                        | 91.2            | 0.99                           | 1.99           | 0.51           |
| benzo(g,h,i)perylene (BghiP) | 9.71             | 11.3                        | 81.5            | 0.99                           | 1.90           | 0.45           |
| indeno(1,2,3-cd)pyrene (InP) | 9.51             | 10.3                        | 85.3            | 0.99                           | 1.91           | 0.53           |
| Min                          | 2.82             | 3.32                        | 81.5            | 0.99                           | 1.05           | 0.30           |
| Max                          | 11.3             | 14.3                        | 100             | 0.99                           | 1.81           | 0.50           |

1.5 min; 195–265 at 8 °C/min; and continued to 315 °C for 1.25 min at 20 °C/min. The MSD was set at 280 °C. Peak verification was based on retention times and target ions of external PAHs. The standard solution for PAHs was prepared using a mixture of 16 US EPA PAHs (Ultra Scientific, North Kingstown, USA) in a concentration of  $500 \pm 0.2 \mu g/mL$ . For internal control, a PAH mixture (lot CH-0209, Ultra Scientific, North Kingstown, USA) was used.

The method precision was evaluated by repeatability using the olive oil fortified with PAH concentrations injected in triplicate (50.0  $\mu$ g/kg, n=20). Accuracy was calculated by recovery. The linearity of the used detector was satisfactory in the range from 5 to 500 mg/kg. The method detection limit (MDL) and method quantification limit (MQL) were defined as signal-to-noise ratios of 3 and 10, respectively.

The average values for precision, reproducibility, recovery, linearity, MQL and MDL are shown in Table 2. They are the same as in our previous work (Kartalović et al., 2022). These results are according to the criteria set by the European Commission Regulation No. 836/2011. Method used in this study was used in PT (FAPAS, 2022) where the z score for benz(a)anthracene (BaA) was 0.1, for chrysene (Chr) 0.4, for benzo(a)pyrene (BaP) 0.8, and for benzo(b)fluoranthene (BbF) 0.3, indicating good results in PAHs measurement. Measurement uncertainties for PAHs complied with the recommendations of Document N°SANTE 11312/2021 (SANTE, 2021).

The data obtained by the experiment were subjected to the analysis of variance (ANOVA) and Post-hoc Duncan test, with significance defined at p < 0.05. Statistical analysis was carried out using Statistica 12.7 (2015, StatSoft Inc., Tulsa, OK, USA).

# **RESULTS AND DISCUSSION**

Fermented sausages (Kulen, Budim sausageslice, Kulen-slice, homemade sausage) contained a casing that was not damaged and fitted well with the filling. On the cross-section, a mosaic composed of approximately uniform pieces of meat and fatty tissue was found. The pieces were evenly distributed and connected to each other. There were no cavities or cracks in them. The samples were stable in color, typical of this type of product. There were no foreign substances or other defects in the samples. The smell and taste were typical for the type of meat and additives. Coarsely ground sausage (Carniolan sausage) has distinguished physical properties without any major folds. The casing fit well with the filling and there was no separated jelly or fat. The meat had a uniform stable color and the filling was evenly distributed and connected. The fatty tissue was whitish and did not fall out of the stuffing when cut. The sausages had a pleasant and distinguishable smell and taste.

The sausages had a specific consistency. There were no foreign substances or other defects in the sample. Smoked products (smoked sirloin with added water, smoked sirloin with added water - slice, dry-cured pork neck with added water - slice) were properly processed, with dry and clean surfaces, without cuts or hanging parts, without a casing or mold on the surface.

The color of muscle tissue was bright red, while fat tissue was white. It had an elastically firm consistency, so the sample chunk could be cut into thin slices. The smell and taste were characteristic, and there were no foreign substances or other defects in the samples. Drycured meat products (dry pork sausage) were properly processed, with dry and clean surfaces, without cuts and hanging parts. There was no casing or mold. The color of muscle tissue was characteristic, bright red, while the fat tissue was white. The product had an elastic-firm consistency, so it could be cut into thin slices.

The smell and taste were pleasant and characteristic. There were no foreign substances or other defects in the sample. Bacon (Kamendin pancetta - slice, homemade bacon) was properly processed, with dry and clean surfaces, without cuts or hanging parts. There was no casing or mold. The color of muscle tissue was characteristic, bright red, and the color of fat tissue was white. The consistency of the product was firm-elastic, but not tough, so the samples could be cut into thin slices. The smell and taste were pleasant and characteristic. There were no foreign substances or other defects in the samples.

The content of 16 US EPA PAH compounds and the sum of PAH4compounds in dry-cured meat products smoked in three different ways (chamber, ATMOS and traditional artisan smokehouse) are shown in Tables 3 - 5.

| РАН     | Homemade sausage | Kulen sausage  | Budim sausage<br>- slice | Kamendin<br>pancetta - slice | Kulen- slice     |
|---------|------------------|----------------|--------------------------|------------------------------|------------------|
| Nap     | < MDL            | $29.62\pm0.17$ | $67.95\pm0.05$           | $4.77\pm0.15$                | $78.20\pm0.2$    |
| Acy     | < MDL            | $14.37\pm0.06$ | $9.83 \pm 0.07$          | $9.39\pm0.17$                | $20.18 \pm 0.05$ |
| Ace     | < MDL            | < MDL          | < MDL                    | < MDL                        | < MDL            |
| Fle     | $43.70\pm0.02$   | $13.50\pm0.07$ | $32.68\pm0.07$           | $39.93\pm 0.29$              | $76.50\pm0.37$   |
| An      | $202.96\pm0.78$  | $46.84\pm0.24$ | $104.04\pm0.41$          | $186.35\pm0.56$              | $263.41\pm0.61$  |
| Ph      | $140.10\pm0.25$  | $30.77\pm0.13$ | $66.92\pm0.09$           | $135.64\pm0.71$              | $173.79\pm0.04$  |
| Fla     | $12.37\pm0.17$   | $2.41\pm0.01$  | $7.18\pm 0.18$           | $12.78\pm0.2$                | $9.64\pm0.1$     |
| BaA     | $7.86\pm0.09$    | $1.32\pm0.01$  | $17.96\pm0.01$           | $9.00\pm0.82$                | $4.39\pm0.12$    |
| Pyr     | $8.65\pm0.02$    | < MDL          | $5.28\pm0.13$            | $7.23\pm0.03$                | $3.92\pm0.39$    |
| Chr     | < MDL            | < MDL          | < MDL                    | $0.82\pm0.07$                | < MDL            |
| BbF     | $0.77\pm0.02$    | < MDL          | < MDL                    | < MDL                        | $0.45\pm0.03$    |
| BkF     | < MDL            | < MDL          | < MDL                    | < MDL                        | < MDL            |
| BaP     | < MDL            | < MDL          | < MDL                    | < MDL                        | < MDL            |
| DahA    | < MDL            | < MDL          | < MDL                    | < MDL                        | < MDL            |
| BghiP   | < MDL            | < MDL          | < MDL                    | $0.85\pm0.05$                | < MDL            |
| InP     | < MDL            | < MDL          | < MDL                    | < MDL                        | < MDL            |
| sum PAH | 4 9.42           | < MDL          | 5.28                     | 8.05                         | 4.37             |

| The content of 16 US EPA PAH com | pounds in meat products | smoked in chambers | $(\mu g/kg \pm SD)$ |
|----------------------------------|-------------------------|--------------------|---------------------|

Table 3.

Results are given as mean  $\pm$  standard deviation (n = 3); naphthalene (Nap); acenaphthylene (Acy); acenaphthene (Ace); fluorene (Fle); anthracene (An); phenanthrene (Ph); fluoranthene (Fla); benz(a)anthracene (BaA); pyrene (Pyr); chrysene (Chr); benzo(b)fluoranthene (BbF); benzo(k)fluoranthene (BkF); benzo(a)pyrene (BaP); dibenz(a,h)anthracene (DahA); benzo(g,h,i)perylene (BghiP); indeno(1,2,3-cd)pyrene (InP); sum PAH4 (sum of benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluranthene and chrysene; method detection limit (MDL)

The value of BaP was below the detection limit in most of the examined products (homemade sausage, Kulen, Kulen-slice, Kamendin pancetta - slice, smoked pork loin with added water - slice, dry-cured pork neck with added water - slice, Carniolan sausage), while the value in both products smoked in a traditional craft smokehouse was 0.84  $\mu$ g/kg (dry pork loin), i.e. 1.1  $\mu$ g/kg (homemade dry bacon).

There is a statistically significant difference (p < 0.05) when it comes to the value of BaP between craft production compared to the values of BaP in products smoked in both a chamber and ATMOS.

Dry-cured homemade bacon had the highest value of PAH4, which was also above the permitted limits according to the current regulations of the Republic of Serbia (Rulebook on maximal limits of contaminant residues in food (2019). No statistically significant difference (p< 0.05) was found in the amount of PAH4 in the samples smoked in the craft smokehouse

and in the chamber, but there is a statistically significant difference between the value of PAH4 in the samples smoked in the craft smokehouses and ATMOS. Dry cured home-made bacon PAH4 value (15.82  $\mu$ g/kg) was higher in comparison with homemade sausage, Kamendin pancetta and Budim sausage PAH4 levels (9.42, 8.05 and 5.28  $\mu$ g/kg) because the smoking method was different. Dry-cured homemade bacon was smoked in a craft smokehouse, while homemade sausage, Kamendin pancetta and Budim sausage were smoked in the chamber.

According to the EU Commission Regulation (2020) No 2020/1255, it was established that despite the application of good manufacturing practices during smoking, it is impossible to achieve lower amounts of PAHs in certain cases of traditionally smoked meat and dry-cured meat products in Ireland, Spain, Croatia, Cyprus, Lithuania, Poland, Portugal, Romania, Slovakia, Finland, Sweden and the United Kingdom. After a detailed evaluation of the gathered information, it was concluded that lo-

|          |                   | bounds in meat products s<br>Smoked pork loin | Smoked pork loin            | ÷ ,                                  |
|----------|-------------------|---|-----------------------------|--------------------------------------|
| РАН      | Carniolan sausage | with added water -<br>chunk                   | with added water -<br>slice | Smoked neck with added water - slice |
| Nap      | < MDL             | $1.04\pm0.01$                                 | $1.67\pm0.04$               | $2.08\pm0.02$                        |
| Acy      | < MDL             | $1.27\pm0.04$                                 | < MDL                       | $0.94\pm0.08$                        |
| Ace      | < MDL             | < MDL   | < MDL                       | < MDL                                |
| Fle      | $7.24\pm0.2$      | $7.80\pm0.1$                                  | $1.75\pm0.04$               | $8.64\pm0.06$                        |
| An       | $21.44\pm0.34$    | $30.60\pm0.19$                                | $8.34\pm0.03$               | $32.14\pm0.19$                       |
| Ph       | $6.01\pm0.05$     | $17.48\pm0.24$                                | $2.87\pm0.05$               | $11.25\pm0.20$                       |
| Fla      | $2.21\pm0.25$     | $\boldsymbol{6.29 \pm 0.03}$                  | $1.12\pm0.13$               | $3.72\pm0.05$                        |
| BaA      | $1.68\pm0.14$     | $5.30\pm0.17$                                 | $0.81\pm0.03$               | $2.84\pm0.03$                        |
| Pyr      | < MDL             | $0.76\pm0.01$                                 | < MDL                       | < MDL                                |
| Chr      | < MDL             | $0.64\pm0.004$                                | < MDL                       | $0.41\pm0.01$                        |
| BbF      | < MDL             | < MDL   | < MDL                       | < MDL                                |
| BkF      | < MDL             | < MDL   | < MDL                       | < MDL                                |
| BaP      | < MDL             | < MDL   | < MDL                       | < MDL                                |
| DahA     | < MDL             | < MDL   | < MDL                       | < MDL                                |
| BghiP    | < MDL             | $0.75\pm0.01$                                 | $0.42\pm0.04$               | < MDL                                |
| InP      | < MDL             | < MDL   | < MDL                       | < MDL                                |
| sum PAH4 | < MDL             | 1.4   | < MDL                       | 0.41                                 |

| Table 4.  |    |
|---|----|
| The content of 16 US EPA PAH compounds in meat products smoked in ATMOS ( $\mu g/kg \pm SE$ | )) |

Results are given as mean  $\pm$  standard deviation (n = 3); naphthalene (Nap); acenaphthylene (Acy); acenaphthene (Ace); fluorene (Fle); anthracene (An); phenanthrene (Ph); fluoranthene (Fla); benz(a)anthracene (BaA); pyrene (Pyr); chrysene (Chr); benzo(b)fluoranthene (BbF); benzo(k)fluoranthene (BkF); benzo(a)pyrene (BaP); dibenz(a,h)anthracene (DahA); benzo(g,h,i)perylene (BghiP); indeno(1,2,3-cd)pyrene (InP); sum PAH4 (sum of benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluranthene and chrysene; method detection limit (MDL)

wer amounts of PAHs cannot be achieved by applying smoking practices within the limits of what is economically feasible and possible without losing specific organoleptic properties of certain traditionally smoked meat and drycured meat products. Therefore, for local production and consumption, a derogation without time limit should be granted for certain traditional smoked meat and dry cured meat products, smoked fish and smoked fish products, namely 5 µg/kg for BaP and 30 µg/kg for PAH4. Given that the products that did not meet the criteria of the Rulebook on maximal limits of contaminant residues in food (2019) were the traditional smoked meat product produced following all the guidelines of good production practice, good hygiene practice and HACCP, there is a need for monitoring of PAH compounds in traditional smoked products, and accordingly, quite likely revision of the existing rulebook for traditional smoked products. The values of BaA, Chr, BbF, BaP were examined in 262 samples of smoked

sausage, smoked bacon, smoked pork "lountza" and smoked ham "chiromeri", grilled pork and poultry produced in Cyprus where 12% of the smoked products and 15% of the grilled meat samples exceeded the permitted values according to EU legislation (Kafouris, Koukkidou, Christou, Hadjigeorgiou & Yiannopoulos, 2020). A group of authors (Mastanjević et al., 2019a) compared the content of PAH compounds in "Slavonian sausage" smoked traditionally and industrially. On that occasion, they showed that the content of the sum of 16 PAH compounds in Slavonian sausage smoked traditionally was 679 µg/kg, while it amounted to 124  $\mu$ g/kg for the sausage smoked in the industrial chamber. It needs to be pointed out that the content of BaP and PAH4 in Slavonian sausage produced in both ways was below the maximum allowed values  $(BaP < 2.0 \ \mu g/kg \text{ and } PAH4 < 9.90 \ \mu g/kg).$ Lorenzo et al. (2011) examined PAHs in smoked samples in the Spain market, actually in two Spanish traditional smoked sausage

Table 5.

| $g/\text{kg} \pm SD$ |                  |                          |
|----------------------|------------------|--------------------------|
| РАН                  | Dry pork loin    | Dry cured homemade bacon |
| Nap                  | < MDL            | $416.31 \pm 1.62$        |
| Acy                  | $3.91\pm0.25$    | $87.51\pm0.59$           |
| Ace                  | < MDL            | < MDL                    |
| Fle                  | $39.15 \pm 0.45$ | $591.50 \pm 0.43$        |
| An                   | $217.11\pm0.87$  | $1313.36 \pm 0.43$       |
| Ph                   | $128.44\pm0.22$  | $886.64\pm0.34$          |
| Fla                  | $18.78\pm0.42$   | $67.05\pm0.43$           |
| BaA                  | $14.87\pm0.10$   | $131.97\pm0.37$          |
| Pyr                  | $0.73\pm0.02$    | $7.19\pm0.62$            |
| Chr                  | $1.21\pm0.02$    | $6.12 \pm 0.43$          |
| BbF                  | $1.10\pm0.01$    | $1.41 \pm 0.43$          |
| BkF                  | $1.22\pm0.15$    | $1.70 \pm 0.43$          |
| BaP                  | $0.84\pm0.05$    | $1.10 \pm 0.45$          |
| DahA                 | < MDL            | $1.79\pm0.42$            |
| BghiP                | $1.23\pm0.12$    | $1.52 \pm 0.37$          |
| InP                  | $0.63\pm0.12$    | $0.58\pm0.02$            |
| sum PAH4             | 3.88             | 15.82                    |

The content of 16 US EPA PAH compounds in meat products smoked in a traditional craft smokehouse  $(ug/kg \pm SD)$ 

Results are given as mean  $\pm$  standard deviation (n = 3); naphthalene (Nap); acenaphthylene (Acy); acenaphthene (Ace); fluorene (Fle); anthracene (An); phenanthrene (Ph); fluoranthene (Fla); benz(a)anthracene (BaA); pyrene (Pyr); chrysene (Chr); benzo(b)fluoranthene (BbF); benzo(k)fluoranthene (BkF); benzo(a)pyrene (BaP); dibenz(a,h)anthracene (DahA); benzo(g,h,i)perylene (BghiP); indeno(1,2,3-cd)pyrene (InP); sum PAH4 (sum of benzo(a)pyrene, benzo(a)anthracene, benzo(b)fluranthene and chrysene; method detection limit (MDL)

varieties: "Chorizo gallego" and "Chorizo de cebolla". The maximum level for BaP in both products was not exceeded in any of the samples – the maximum level in that period was 5  $\mu$ g/kg, but it did not exceed the current maximum level. BaP for "Chorizo de cebolla" was 0.88  $\mu$ g/kg, and 0.65  $\mu$ g/kg for "Chorizo gallego".

PAH4 and BaP were below the limit of detection in all examined samples of Petrovská klobása, smoked in traditional and industrial conditions (Škaljac et al., 2014). Slavonska ham PAH4 concentrations were above the legislative recommendation (>  $30 \mu g/kg$ ) in seven samples (Mastanjević et al., 2019b). Puljić et al. (2019) evaluated the difference in PAH content in samples of traditional dry-cured pork meat products, "Hercegovačka pečenica", produced in a traditional smoke-house and industrial chambers.

The value of PAH4 in the samples produced in a traditional smoking way highly exceeded the

maximum set limits (12  $\mu$ g/kg, which is up to 10 times).

#### CONCLUSIONS

This study showed that the values of PAH compounds in smoked meat products in controlled industrial ways (ATMOS and chambers) are within the limits proscribed by national and European legislation.

PAH4 value for one of the two products traditionally smoked in craft smokehouses was above the prescribed limit set at the national level.

The fact that the product that did not meet the criteria of the current Rulebook (2019) was a traditionally smoked meat product made in compliance with all the guidelines of good production practice, good hygiene practice and HACCP, means that there is a need for monitoring of PAH compounds in traditionally smoked products and quite likely a revision of

the existing rulebook on traditionally smoked products according to EU practice (Regulation of the EU Commission 2020/1255).

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### POLICIKLIČNI AROMATIČNI UGLJOVODONICI KAO LIMITIRAJUĆI PARAMETAR TRADICIONALNE PROIZVODNJE SUHOMESNATIH PROIZVODA

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> Sažetak: Cilj studije bio je da se ispitaju koncentracije 16 US EPA policikličnih aromatičnih ugljovodonika (PAH-ova) i provere senzorna svojstva kod 11 suhomesnatih proizvoda, dimljenih na tri različita načina (komora za simulaciju atmosfere (ATMOS), industrijska komora i pušnica za tradicionalno zanatsko dimljenje) u jednoj industriji mesa u Srbiji. Kontrolisanom industrijskom proizvodnjom se ne mogu postići sva ona prepoznatljiva i karakteristična senzorska svojstva tradicionalnih dimljenih proizvoda, pa se sve češće i industrije mesa opredeljuju da u svoj program uključe i proizvode dimljene u tradicionalnim zanatskim pušnicama. Metodom gasne hromatografije-masene spektrometrije (GC-MS), analizirano je 16 US EPA PAH-ova. Dobijeni rezultati su deo procesa validacije tehnologije proizvodnje testiranih suhomesnatih proizvoda. Ukupne koncentracija benzo(a)pirena, benzo(a)antracena, benzo(b)flurantena i hrizena (koji čine takozvanu "PAH4" grupu) u tradicionalnoj domaćoj dimljenoj slanini proizvedenoj u skladu sa svim smernicama dobre proizvodnje i higijenske prakse, dostigla je vrednost od 15,82 µg/kg, što je bilo iznad legalne granice propisane nacionalnim zakonodavstvom. Ovo ukazuje na potrebu za monitoringom sadržaja PAH jedinjenja u tradicionalnim dimljenim proizvodima, a shodno tome i revidiranje postojećeg pravilnika za tradecionalne dimljene proizvode u skladu sa praksom EU.

Ključne reči: PAH, mesni proizvodi, tradicionalna proizvodnja, ATMOS

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