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## OCURRENCE OF POLYCYCLIC AROMATIC HYDROCARBONS IN BOTTOM ASH FROM INDIVIDUAL HEATING DEVICES

### Abstract

Combustion of solid fuels such as coal, biomass and, contrary to the applicable law, waste in individual heating devices still remains a serious problem in Poland. It causes the generation of large amounts of pollutants and harmful substances contained not only in fly ash released into the air with smoke but also in the bottom ash, which constitutes a serious environmental problem. This paper presents the results of a comparative study on the contents of 16 polycyclic aromatic hydrocarbons (PAHs) in bottom ash from the combustion of hard coal (HC), wood (W) and a mixture of different solid fuels including municipal waste (MW), their sums and profiles. For the bottom ash samples taken for these fuels, the share of carcinogenic congeners in the sum of PAHs, toxicity, mutagenicity and carcinogenicity equivalents have also been determined. The highest content of total PAHs, amounting to an average of 20.7 ppb, was recorded for bottom ash obtained from combustion of a mixture of solid fuels and waste. For such ash, the toxicity and carcinogenicity equivalents were approximately twice as high as for other types of ash. Bottom ash was found to be dominated by benzo[a]anthracene, and the tetracyclic congeners accounted for 60-68% of all PAHs (W<MW<HC). Regardless of the origin of the bottom ash, approximately 60% of the total PAHs were constituted by carcinogenic compounds.

**Keywords:** solid fuel combustion, ash, gas chromatography, PAHs, harmfulness

## WYSTĘPOWANIE WIELOPIERŚCIENIOWYCH WĘGLOWODORÓW AROMATYCZNYCH W POPIOŁACH PALENISKOWYCH POCHODZĄCYCH Z INDYWIDUALNYCH URZĄDZEŃ GRZEWCZYCH

### Abstrakt

Spalanie paliw stałych, m.in. węgla, biomasy, a także, mimo zakazu, odpadów komunalnych w indywidualnych systemach grzewczych stanowi w Polsce nadal poważny problem. Powoduje to powstawanie dużych ilości zanieczyszczeń i szkodliwych substancji zawartych w pyłach, które trafiają wraz z dymem do powietrza bez jakiegokolwiek kontroli, a popiół paleniskowy będący odpadem stanowi poważny problem środowiskowy. W pracy przedstawiono wyniki badań porównawczych zawartości 16 wielopierścieniowych węglowodorów aromatycznych (WWA) w popiele paleniskowym pochodzącym ze spalania węgla kamiennego (HC), drewna (W) oraz mieszaniny różnych paliw stałych, w tym odpadów komunalnych (MW), ich sumy oraz profile. Wyznaczono także udział kancerogennych kongenerów w sumie WWA, równoważniki toksyczności, mutagenności i kancerogenności. Największą zawartość sumy WWA, wynoszącą średnio 20,7 ppb, odnotowano dla popiołu paleniskowego pochodzącego ze spalania mieszaniny paliw stałych i odpadów. Dla tego popiołu równoważniki toksyczności i kancerogenności były około dwa razy wyższe niż dla pozostałych popiołów. W popiołach dominował benzo[a]antracen, a czteropierścieniowe kongenery stanowiły 60–68% wszystkich WWA ( $W < MW < HC$ ). Niezależnie od pochodzenia popiołu paleniskowego ok. 60% sumy WWA stanowiły związki kancerogenne.

**Słowa kluczowe:** spalanie paliw stałych, popiół, chromatografia gazowa, WWA, szkodliwość

### 1. Introduction

A significant source of pollutants introduced into air in Poland is constituted by low-stack emission generated by local heating systems, domestic heating appliances and transport. The quantity and type of substances emitted into the air from low-stack sources strongly depends on the share of solid fuels combusted in households for heating purposes. Based on the CSO 2020 report [1], in 2018 almost half of the households in Poland, i.e. 45.4%, have been using heating appliances with solid fuels, of which dual-function central heating boilers (generating heat energy and hot water), were the most common (42.2% of households). Single-function boilers, on the other hand, were used by 39.0% of households. Almost all boilers belonging to these 2 types constitute primary appliance in a household (98.3% of dual-function and 96.5% of single-function boilers). It should be noted that in Poland, 13.7% of households still use the most traditional heating devices, i.e. furnaces in rooms, mainly tiled stoves, of which as many as 91.8% are the only heating devices, while the rest of them are used occasionally. About 10% of households use fireplaces, usually of the closed type, among which they are the only source of heat in 12.4% of households. The low height of the emitters favours the direct impact of combustion pollutants on people and the environment.

Boilers with solid grates burning solid fuels and, despite the ban applicable in Poland, sometimes also municipal waste [2–4], fail to assure combustion conditions in which no harmful substances are formed as products of incomplete combustion. In particular combustion temperatures that are too low, as is often the case with domestic boilers, cause toxic gas and dust emissions. The finer dust fractions with particle diameters from 0.5  $\mu\text{m}$  to 100  $\mu\text{m}$  [5] emitted into the atmosphere with no kind of abatement, comprise fly ash (FA), while the coarser particles with diameters of 0.125–2 mm enter the bottom ash (BA) [5]. The formed dust contains a significant amount of soot, which is a carrier of toxic and carcinogenic substances into the environment [6]. Due to its adsorbent properties, bottom ash can contain persistent organic compounds including polycyclic aromatic hydrocarbons (PAHs) [2, 4, 7], dioxins and furans (PCDD/Fs) [8–12] derived from the source material or formed during combustion, but also hazardous metal compounds [12–15]. In Poland, household ash is most often dumped in landfills. Sometimes it is also disposed of in fields and gardens, as there is a misconception about their beneficial fertilising properties [13]. Both dusts derived from household and also from waste incineration plants or the energy sector, represent a serious environmental problem [14, 16]. For example, in 2018, almost 19 Mt of bottom ash was produced at waste incineration plants in Europe alone, while only 46% of this material was treated in any way [17, 18]. Consequently, research is being conducted worldwide on the use of combustion by-products, such as fly ash, bottom ash and boiler slag [16, 19–22], particularly in the construction and ceramics industries [21, 23, 24]. Hence, the work of many researchers has mainly been focused on inorganic components in waste dusts [13, 14, 16, 23, 24], and there is a lack of literature reports on organic components, especially in real samples. The aim of this work was therefore to identify the content of harmful persistent organic compounds such as polycyclic aromatic hydrocarbons present in actual bottom ashes, the proportions in which they occur and their harmfulness depending on the fuel burned. To determine harmfulness, use was made of indicators such as toxicity factor TEQ (*Toxic Equivalent*), mutagenicity MEQ (*Mutagenic Equivalent*) and carcinogenicity TCDD-TEQ (*Carcinogenicity Equivalent*) [25–29].

## 2. Materials and methods

The subjects of the study were bottom ash samples coming from small power boilers (up to 18 KW), where hard coal (HC), wood (W) or a mixture of different solid fuels, including municipal waste (MW), was burned. Representative ash samples weighing approximately 0.5 kg were collected in the Lesser Poland Region and Silesian province (Fig. 1). They differed mainly in colour (light grey or grey) and granulation. In the dust from the wood-fired boilers, fragments of incinerated

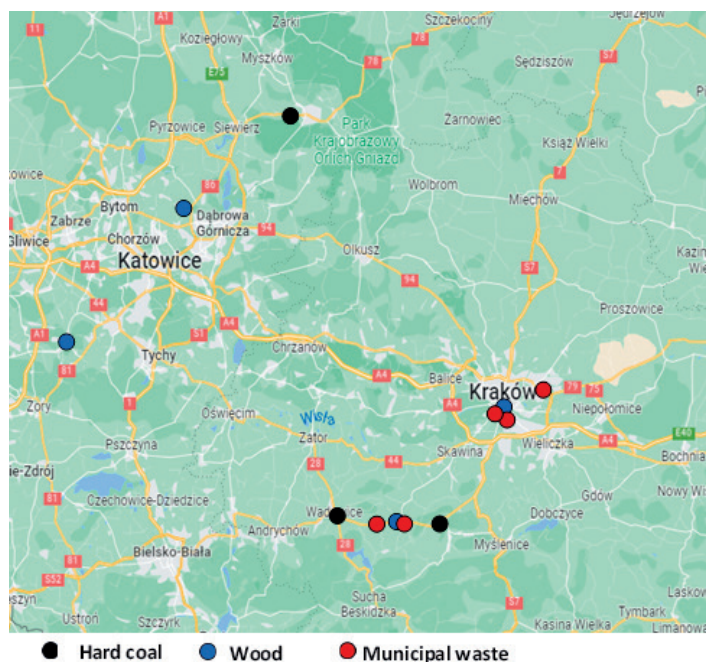


Fig. 1. Location of the collection points for bottom ashes from domestic heating stoves (map data: 2022© Google, ORION-ME)

biomass were present. Each dust sample was sifted through a sieve with a mesh diameter of 2 mm. Approximately 10 g of the tested sieved ash was weighed on an analytical balance (RADWAG, Poland) to the accuracy of 0.1 mg and was carried out for a period of 60 minutes extracted with dichloromethane (Sigma-Aldrich) in an ultrasonic bath. The extract was filtered and evaporated until dry. Then it was dissolved in toluene (Sigma-Aldrich), obtaining a test sample with a volume of 0.1 cm<sup>3</sup>. The extract from the bottom ash samples was chromatographically analysed for the 16 priority PAHs using a Clarus 500 gas chromatograph (Perkin Elmer, USA) equipped with an autosampler and a flame ionization detector (FID). The compounds were separated on a capillary column (Restek RTX-5, 5% phenyl methyl siloxane, 30 m × 0.32 mm × 0.25 μm film). Helium was used as the carrier gas with a flow rate of 1.5 cm<sup>3</sup>/min. The 3 μl samples were introduced using split/splitless injection, with the temperature of the injector at 240°C. The detector temperature was 280°C. For the PAH analysis, the initial temperature of the oven (60°C) was held for 4 min, after which the temperature grew at 10°C/min to 280°C, and then was held for 14 minutes. The FID detector was supplied with air (450 cm<sup>3</sup>/min) and hydrogen (45 cm<sup>3</sup>/min), the FID's temperature was 280°C. In the bottom ash, 16 priority PAHs were determined, viz.: naphthalene (Na),

acenaphthylene (Acy), acenaphthene (Ace), fluorene (Flu), phenanthrene (Fen), anthracene (An), fluoranthene (Flt), pyrene (Py), benzo[a]anthracene (BaA), chrysene (Ch), benzo[b]fluorene (BbF), benzo[k]fluorene (BkF), benzo[a]pyrene (BaP), indeno[1,2,3-cd]pyrene (IP), dibenzo[a,h]anthracene (DBA) and benzo[g,h,i]perylene (BghiP).

Based on the predetermined calibration curves describing 16 standard PAHs, quantitative determination has been carried out. The linear correlation between the peak surface areas and PAH concentrations was checked within the range 1–10 ng/μl (using Ultra Scientific (USA) PAH Mix PM-611 with a concentration of 100 μg/ml for each PAH in dichloromethane). The correlation coefficients for this concentration range for the 14 PAHs were 0.99 and for BbF and BkF 0.97, and the limit of detection (LOD) was 0.01 ppb for Fen; 0.03 ppb for BbF, BkF and DBA for the other congeners 0.02 ppb. The limit of quantification (LOQ) for individual PAHs ranged from 0.03 ppb to 0.09 ppb.

### 3. Results and discussion

Polycyclic aromatic hydrocarbons in fly ash and bottom ash are products of inefficient combustion, and their concentrations and profiles are strongly associated with fuel properties, furnace type and combustion conditions [30–32]. 16 priority polycyclic aromatic hydrocarbons were identified in samples of bottom ash from households burning coal, wood and a mixture of different fuels, including municipal waste. The mean concentrations of total PAHs for HC and W were similar at 10.2 ppb, while for MW it was found to be two-fold higher (20.7 ppb) (Table 1). Benzo[a]anthracene was dominant in all samples. Its average percentage for HC was 51.5% and for W and MW between 42% and 43% (Fig. 2). Naphthalene and acenaphthylene were not detected in the dust from coal combustion, similarly to the study by Fabiańska and Smółka-Danielowska [33]. Only wood combustion dust was found to contain naphthalene at 0.14 ppb, while the average content of acenaphthylene in wood combustion and municipal waste dust was 0.06 ppb and 0.12 ppb, respectively. In addition to benzo[a]anthracene (42–51%), phenanthrene, fluoranthene, fluorene and pyrene were most abundant in the dusts studied (totaling from 35.5 to 36.2%). The average content of PAHs congeners in bottom ash was in the following order: for hard coal – BaA > Fen > Flt > Flu > Ch > An > BbF > BaP > BkF > IP > Py > DBA > BghiP; for wood – BaA > Flu > Fen > Flt > Ch > Ace > BbF > Py > BaP > An > BkF > Na > DBA > IP > Acy > BghiP and for a mixture of diverse raw materials – BaA > Fen > Flt > Flu > Ch > Py > BbF > An > BkF > BaP > Ace > DBA > Acy, IP > BghiP. In bottom ash, irrespective of the initial raw material burned, benzo[g,h,i]perylene was found to be the least abundant of the PAHs determined.

Table 1. Mean content (min.-max. values) of the 16 PAHs congeners,  $\Sigma$ PAHs and mean values of the exposure indicators (PAH<sub>canc</sub>/ $\Sigma$ PAHs, TEQ, MEQ and TCDD-TEQ) for a mixture of PAHs in bottom ashes

	Coal (C)	Wood (W)	Municipal waste (MW)
ppb			
Na		0.14 (0.04-0.24)	
Acy		0.06 (0.05-0.07)	0.12 (0.05-0.18)
Ace	0.08 (0.03-0.11)	0.56 (0.12-1.09)	0.43 (0.14-0.86)
Flu	0.73 (0.18-1.17)	1.24 (0.56-2.29)	2.10 (1.13-3.28)
Fen	1.36 (1.17-1.67)	0.93 (0.78-1.11)	2.27 (1.22-4.11)
An	0.29 (0.23-0.37)	0.25 (0.08-0.40)	0.60 (0.22-1.82)
Flt	0.99 (0.97-1.01)	0.81 (0.28-1.35)	2.10 (0.44-5.37)
Py	0.09 (0.07-0.11)	0.28 (0.16-0.41)	0.98 (0.14-4.00)
BaA	5.30 (4.21-6.70)	4.5 (3.67-5.49)	8.88 (3.86-16.68)
Ch	0.65 (0.51-0.76)	0.75 (0.38-1.07)	1.08 (0.92-1.18)
BbF	0.26 (0.19-0.38)	0.31 (0.09-0.57)	0.81 (0.08-1.38)
BkF	0.15 (0.10-0.23)	0.18 (0.11-0.31)	0.60 (0.14-1.54)
BaP	0.18 (0.05-0.34)	0.26 (0.07-0.52)	0.55 (0.05-2.39)
IP	0.10 (0.08-0.12)	0.07 (0.04-0.13)	0.11 (0.05-0.20)
DBA	0.08(0.07-0.10)	0.12 (0.09-0.14)	0.19 (0.06-0.51)
BghiP	0.03 (0.02-0.03)	0.04 (0.03-0.06)	0.07 (0.03-0.14)
$\Sigma$ PAHs	10.26 (9.29-11.36)	10.25 (8.23-12.67)	20.69 (11.25-38.17)
PAH <sub>canc</sub> / $\Sigma$ PAHs	0.65 (0.56-0.72)	0.60 (0.55-0.71)	0.58 (0.44-0.66)
TEQ	6.52 (5.25-7.96)	6.03 (4.81-7.16)	11.91 (6.86-22.68)
MEQ	0.76 (0.53-0.92)	3.31 (1.04-4.67)	4.22 (0.63-4.37)
TCDD-TEQ <sup>*)</sup>	2.02 (1.87-2.11)	2.34 (1.33-2.84)	5.52 (0.88-12.84)

<sup>\*)</sup> ppt

PAH<sub>canc</sub>/ $\Sigma$ PAHs = ([BaA]+[BaP]+[BbF]+[BkF]+[Ch]+[DBA]+[IP])/([ $\Sigma$ PAHs]) [36,37]

TEQ = 0.001×([Na]+[Acy]+[Ace]+[Flt]+ [Fen]+[Flu]+[Py])+0.01×([An]+[Ch]+[BghiP])+0.1×([BaA]+[BbF]+[BkF]+[IP])+1×[BaP]+5×[DBA] [25]

MEQ = 0.00056×[Acy]+0.082×[BaA]+0.017×[Ch]+0.25×[BbF]+0.11×[BkF]+1×[BaP]+0.31×[IP]+0.29×[DBA]+0.19×[BghiP] [26,28]

TCDD-TEQ=0.000025×[BaA]+0.00020×[Ch]+0.000354×[BaP]+0.00110×[IP]+0.00203×[DBA]+0.00253×[BbF]+0.00487×[BkF] [27,28]

Source: own study

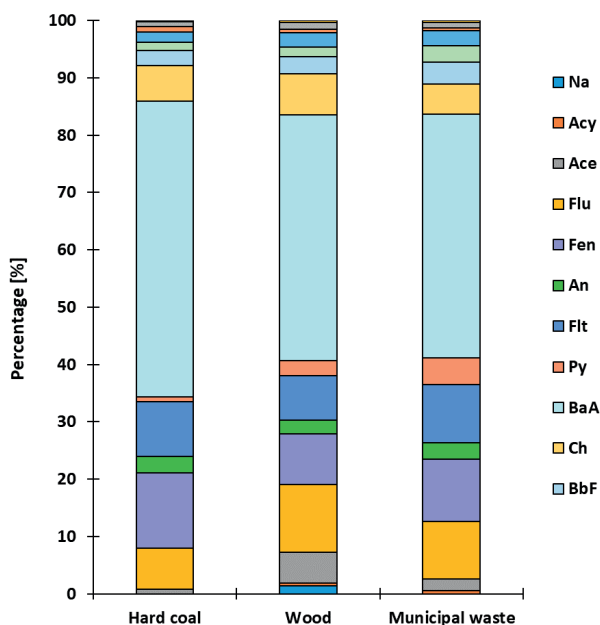


Fig. 2. Average percentage of PAHs congeners in combustion waste from burning hard coal, wood and mixture of different solid fuels

Source: own study

The greatest differences in concentrations of individual PAHs congeners were recorded for bottom ash samples from the combustion of a mixture of different solid fuels, including municipal waste. For these samples, PAHs ranged from 11.2 ppb to 38.2 ppb, while for coal combustion ash from 9.29 ppb to 11.36 ppb. In the case of real samples, especially where waste is burned, it is difficult to determine the composition of the mixture used for combustion, hence such differences in concentrations for individual samples. As a rule, in the publications authors present the results of experimental studies where they burn mixtures of different solid fuels of a strictly defined composition and conduct the combustion under controlled conditions [3, 4, 31–34]; the same is true for the combustion of coal or the co-firing of coal with biomass in power plants [29, 35].

Considering the number of benzene rings in the molecule, all ashes are dominated by 4-rings PAHs, i.e. Flt, Py, BaA, Ch. Their content in total PAHs comes up to 60–68%, with the highest percentage recorded for HC ash (HC>MW>W) – Fig. 3. Similarly, Fabiańska et al. reported the content of 4-rings PAHs in a total of 16 PAHs at 64–80% in fly ash from coal combustion in power plants and in boilers with mechanical or fluidised bed grates [35]. For the other congeners, the proportion of 2- and 3- rings ones is highest for W dust (30.3%) and 5- and 6-rings for MW dust (11.2%).

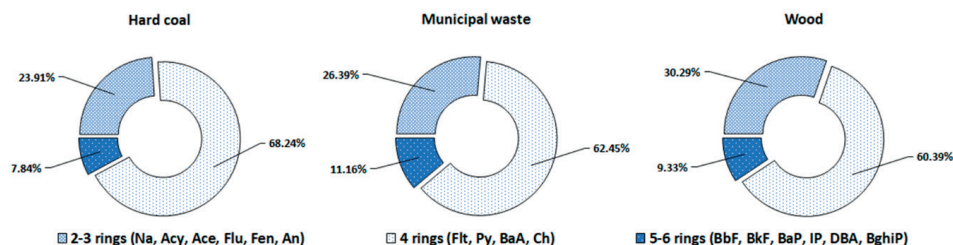


Fig. 3. Profiles of PAHs in bottom ashes from domestic heating furnaces

Source: own study

In order to determine the harmfulness of PAHs contained in combustion dust, the proportion of so-called carcinogenic PAHs in the total of all PAHs determined was calculated ( $PAH_{\text{canc}}/\Sigma PAHs$ ) and equivalents TEQ, MEQ and TCDD-TEQ (Table 1) described in detail in literature [25-29,36]. Regardless of the origin of the bottom ash, approx. 60%  $\Sigma PAHs$  are carcinogenic compounds. Similar results were obtained for fly ash from coal combustion in power plants, in water and steam boilers with mechanical grates, in dust emitted from coking plants [29], in soot samples [34] as well as in the particulate matter of cities in the Silesian region [38-40]. Lower values of around 0.4 to 0.5 were recorded for fly ash from co-firing coal with forest biomass in a fluidised bed boiler and dust emitted from internal combustion engines [29]. The toxicity equivalent TEQ for MW dust was approximately twice as high as for the other dusts tested, at 11.9 ppb. Very similar is the case of TCDD-TEQ, where for the individual dusts the equivalent was 2.0 ppt (HC), 2.3 ppt (W) and 5.5 ppt (MW). The situation was different for MEQ. The lowest mutagenicity index of 0.8 ppb was observed for bottom ash from coal combustion, while for W and MW dust the values were 2.3 ppb and 5.5 ppb, respectively (Table 1). In general, TEQs, MEQs and TCDD-TEQs for the studied dusts were found to be 100-fold or much lower than those for fly ash of various origins [29] or for particulate matter in Poland (own studies).

#### 4. Summary and conclusions

In Poland, almost half of all households still use individual heating appliances fuelled by solid fuels, which are the main source of atmospheric emissions of combustion-characteristic pollutants. Primarily burnt is hard coal, characterised by an ash content of up to 12% [41], as well as biomass, and unfortunately, despite the ban, municipal waste. The waste product is in this case constituted by significant amounts of bottom ash, which is mainly deposited in landfills. Its constituents may penetrate the soil, or get into the air in a way of secondary dusting. The composition



of bottom ash derived dust is therefore important, particularly with regard to hazardous substances. The study analysed dust obtained in combustion of different solid fuels (hard coal, wood, mixing of different fuels), with particular focus on polycyclic aromatic hydrocarbons. On the basis of the study, it was concluded that:

- In the ashes studied, the content of the sum of 16 PAHs ranged from 8.32 ppb to 38.17 ppb. Among the PAHs congeners, benzo[a]anthracene dominated (42-51%), which translates into more than 60% share of carcinogenic PAHs in their total, as well as a high proportion of 4-rings PAHs (60-68%).
- The harmfulness of the dusts, characterised by equivalents of toxicity, mutagenicity and carcinogenicity, indicates that the most hazardous of the bottom ashes tested are those coming from boilers of low power, where municipal waste is burned.
- The equivalents determined in the work have overall small values: TEQs ranging from 4.8 ppb to 22.7 ppb and MEQs ranging from 0.5 ppb to 4.67 ppb; in the case of TCDD-TEQs its values range from 0.9 ppt to 12.8 ppt and are at least 100 times lower than for other dust origins, i.e. fly ash, emitted dust or particulate matter.
- In general, the content of the 16 priority PAHs in the actual bottom ash produced by low power boilers is much lower than in other types of dust, but this does not mean that these compounds should be ignored in the assessment of harm to human health and the environment.

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