VÁCLAV VESELÝ[1], ZBYSZEK SZELIGA[2], ZUZANA VÁVROVÁ[2],
BOHUMÍR ČECH[2], PAWEŁ REGUCKI[3], RENATA KRZYŻYŃSKA[4]

CHARACTERISTIC OF MERCURY ON THE SURFACE OF ASH ORIGINATING FROM ELECTROSTATIC PRECIPITATORS OF LIGNITE AND BITUMINOUS COAL-FIRED POWER PLANTS

Mercury concentrations in ash taken from electrostatic precipitations (ESPs) installed in bituminous coal and lignite power plants have been analyzed by the X-ray fluorescence (XRF) method and leaching test supported by detailed coal and ash compositions' analyses, surface scans and particles size distribution studies. The results showed that mercury was present on the surface of ash particles. Its concentration decreased upon increasing size of ash particles. Leaching tests showed that only small part of mercury was removed with water which suggests the fact that it occurred mostly in the form of insoluble compounds such as Hg, HgO, HgS and Hg_2Cl_2. There existed ash particles of the diameters from 50 to 60 μm, characterizing by the maximum capability to adsorb mercury and its compounds. The authors suggest that metals like copper and lead formed ash active centers that were preferably occupied by molecules containing atoms of mercury. It was highly possible to expect that content of these elements in ash would have a significant effect on sorption of mercury from combustion gases.

1. INTRODUCTION

Mercury, emitted to the environment mainly during coal combustion, is a very strong neurotoxin for humans and the environment. For these reasons the mercury emission is carefully controlled and the respective standards lead steadily to its reduction [1]. The concentration of mercury in fossils depends on its type and locality [2]. In Czech

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[1]Institute of Chemical Process Fundamentals, Academy of Sciences of the Czech Republic, Prague, Czech Republic.

[2]VŠB Technical University Ostrava, CENET, Centre of Energy Utilization of Non-traditional Energy Sources Ostrava, Czech Republic.

[3]Wrocław University of Science and Technology, Faculty of Mechanical and Power Engineering, Wrocław, Poland.

[4]Wrocław University of Science and Technology, Faculty of Environmental Engineering, Wrocław, Poland, corresponding author R. Krzyżyńska, e-mail address: renata.krzyzynska@pwr.edu.pl

Republic, as well as in Poland, lignite is an important part of combusted fuels. The concentration of mercury in this lignite was analyzed by Pilar et al. [3]. As a result of combustion process, the particles of ash are formed in the furnace. Regardless of power unit types, ash occurs in two forms, one type is removed from the combustion chamber as slag and the other one is fly ash which is taken by exhaust gases from the furnace up to environment. Mercury presents in fossil is firstly evaporated and next, passing to the stream of exhaust gases, follows the same way as particles of fly ash which are transported to electrostatic precipitations. During the flow through pipes, heat exchangers and various auxiliary units enabling separation of harmful compounds, the exhaust gases are steadily cooled down and, at the same time, impurities are removed in individual separation units.

Cyclones, electroseparators, various filters or their combinations are used for separation of fly ash. Acidic components are separated by various processes such as wet, semidry and dry chemisorption methods [4]. Auxiliary units that enable separation of undesirable pollutants cause changes in composition of exhaust gases [5]. The compounds which are used in the purifying processes are catalytically active, very often selective ones or can react as sorbents. They contain, e.g., oxides of vanadium, tungsten, iron or copper. Sorbents are very often used in the form of solid compounds based on carbon, inclusive its combination with calcium salts or zeolites. The aim of these approaches is dealing with the following problems:

• reduction of the combustion products to the level satisfying the existing emission limits (ash, SO_x, HCl, NO_x, etc.),

• examination of the installed unit (SCR [6], ESP or FGD [7]) on elimination or reduction of mercury emission.

In typical, conventional power plants exhaust gases from the combustion chamber enter the electrostatic precipitator and pass through a labyrinth of electrodes. Electroseparators operate on the principle of attractive forces acting between the electrically charged ash particles and the electrodes having the opposite charge. Fly ash particles are charged by ionized gas that passes through the electrostatic field. Corona is formed between the electrodes where solid particles of ash move towards the electrodes and precipitate where voltage on electrodes is from 30 to 80 kV. Separated ash is collected in hoppers of individual sections which are formed by a bundle of electrodes when exhaust gases freely pass through the chambers. The number of sections of electrostatic filter is determined by design of the power plant and properties of ash. Block diagram of the three ESP chambers is given in Fig. 1.

The efficiency of ash particles separation in ESP is higher than 99%. The rest, negligible part of ash is emitted into environment with purified gases. The velocity of gas inside the EPS unit is from 0.15 up to 0.62 m/s and is a multiple of terminal velocity of ash particles. The calculated terminal velocities of ash change from 10^{-5} m/s for particle diameters equal to 1 μm up to approximately 0.1 m/s for particle diameters equal to 100 μm [8].
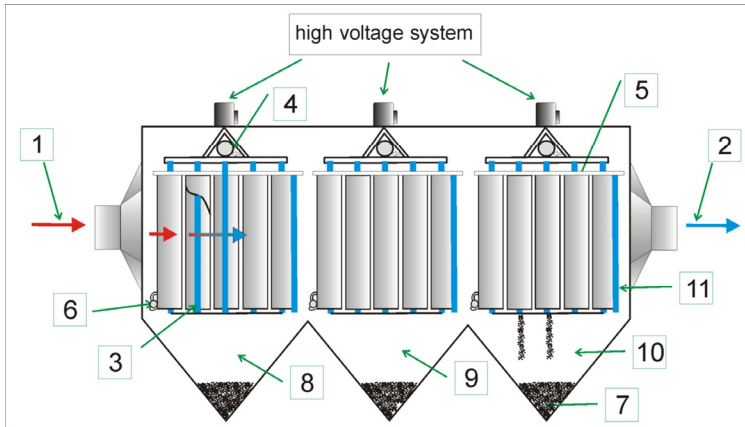
Fig. 1. Block diagram of an electroseparator: 1 – inlet of polluted exhaust gases, 2 – outlet of purified exhaust gases, 3 – electrodes, 4 – hammering of electrodes, 5 – precipitating electrodes, 6 – hammering of precipitating electrodes, 7 – separated ash, 8 – first section/hopper ESP, 9 – second section/hopper ESP, 10 – third section/hopper ESP, 11 – baffles (partition walls)

Large ash particles are separated from the exhaust gas in the first chamber, while fine particles are separated in the last chamber. The amount of separated fly ash from lignite in the first chamber achieves 85–89% of total ash mass. In the second chamber, 10–12% of total ash and in the third (last) one 1–3% is separated. To achieve similar separation efficiency of fly ash from bituminous coal, four ESP chambers are necessary.

Zhang et al. report [9] that the content of Hg^{2+} in exhaust gases entering ESP in some Chinese energy power plants has a significant effect on adsorption of mercury on fly ash. Electroseparators separate from 10 to 30% of the total mass of mercury in exhaust gases. Ash separated in the individual chambers varies in the particle size distribution depending on the type of combusted coal, its processing technology and combustion regime.

Due to its very high separation efficiency, fly ash could be used as a sorbent of potential harmful flue gas components. Separated ash particles are relatively small and have a specific surface in the range from 2 to 5 m^2/g what means that they cannot be treated like typical sorbents, for which specific surface is in the order of hundreds of m^2/g. However, they can be used for adsorption of heavy metals like mercury. Two types of forces affect surface adsorption between the gas and ash particles, and thus two types of adsorption are recognized:

• Physical adsorption based on weak van der Waals forces. These are weak forces resulting from interactions between induced, permanent or transient electric dipoles acting on small distances.

• Chemical adsorption with covalent bonds between gas molecules and solid adsorbent.

Wilcox et al. in [10] distinguish between two basic forms of ash. The former one is formed as a product of hard coal combustion. It is non-self-cementing and contains SiO_2, Al_2O_3, Fe_2O_3 as well as a small amount of CaO. The content of the compounds is higher than 70% of its total mass. The other type of ash, generated by lignite, has different composition and contains alkaline compounds. Its melting point is relatively low. Querol et al. in [11] identified elements that are present both in slag and fly ash. Volatile elements like As, B, Bi, Cd, Ge, Hg, Mo, Pb, S, Se, Sb, Sn and Tl are mostly present in fly ash. It is interesting that concentration of mercury adsorbed on fly ash is ten times higher than that in slag.

Querol et al. in [10] also reported that particles smaller than 10 μm adsorb the abovementioned elements including mercury. Volatile elements are associated first of all with chlorides, sulfates and carbon. With regard to their volatility these elements evaporate in the combustion chamber (above 1000 °C) and after cooling down in ESP (at 150–200 °C) condensate on particles of fly ash. According to correlations given by Zhang et al. [9], the maximum efficiency of mercury adsorption on ash in ESP equals 39%. The size of particles, their porous structure and ash composition have a significant effect on the amount of mercury adsorbed on ash particles [12, 13]. An important influence on the sorption properties of ash has the quantity and structure of unburned carbon [14] which is controlled by the combustion regime. A significant effect on the adsorption of mercury on ash particles has also the content of chlorine in coal. Chlorine oxidises metallic mercury forming Hg^{2+} compound which is adsorbed on unburned carbon in ash [15].

With regard to a considerable variation of the coal composition, the adsorption of pollutants, including mercury, on ash could not be precisely determined. Quantity and quality of pollutants adsorbed on ash are usually evaluated by leaching. Mercury, as one of the studied pollutants, belongs to the group of compounds differing by their solubility in water. To the first group of compounds belongs the insoluble mercury dichloride ($HgCl_2$) and mercury sulfate ($HgSO_4$), whereas to the other group – elementary mercury (Hg), mercury oxide (HgO) and calomel (Hg_2Cl_2). Properties of some selected compounds are given in Table 1.

Table 1

Physical properties of selected mercury compounds [16]

Analyte	Molecular weight [g/mol]	Melting point [°C]	Boiling point [°C]	Solubility at 25 °C [g/dm³]
Hg	200.58	−38.8	356.7	insoluble
HgO	216.58	500	decomposes	5×10^{-2}
HgS	232.66	583	decomposes	1×10^{-5}
$HgSO_4$	296.65	–	sublimes at 450 °C	decomposes
$HgCl_2$	271.50	276	302	69
$HgBr_2$	360.41	236	322	6.1

Mercury readily forms amalgams with Au, Ag, Cu, Zn, Cd, K, Pb and Na, while with metals of the iron-type such as Fe, Ni and Co no amalgams are formed. Ash mostly consists of the mixture of oxides, not pure metals and this is the reason why amalgams do not form. These elements or their compounds could activate the surface of ash and thus form active centers for adsorption of mercury compounds including atomic mercury. The main influence on the adsorption of mercury on ash particles has the amount of unburned carbon and its form, the amount of mercury ions in exhaust gases and chemical composition of ash.

2. EXPERIMENTAL SETUP AND METHODS

Chemical and physical properties of investigated fossil fuels. The lignite originated from the mine in Krušné Mountains coalfield and was collected in two power plants (PPI and PPII). Samples (ca. 1 kg each) were collected every day for the period of three weeks. Next, they were mixed together, homogenized and the proximate and elemental analyses of an average sample of lignite were made. Four samples of bituminous coal from the Silesian field were collected per week and were mixed and analyzed in the same way as lignite samples. The average composition of both fuels, characterized by the content of C, H, N, S and ash in the dry matter, was made according to the European analytical standards.

In order to verify the size and shape of ash particles collected in individual ESP chambers, the authors made a series of tests using the SEM technique based on scanning electron microscope Tescan Indusem equipped with a Quantax 200 and XFlash detector 5010 instruments from Bruker for energy-dispersive X-ray spectroscopy (EDX) at accelerating voltage of 15 kV, that produces images of a sample by scanning the ash surface with a focused beam of electrons. Bulk weight of fly ash has been determined experimentally by the porosimetry (AccuPycII 1340 V1.05) and changes from 1102 up to 1292 kg/m^3. The characteristic diameter of particles of the given ash has been determined using a particle size analyzer (ANALYST 22) by the method described elsewhere [17].

Mercury concentration in bituminous and lignite coals. The samples of ash were decomposed in aqua regia and analyzed by the atomic absorption spectroscopy (AAS) method (Perkin Elmer, AVIO 500) to determine the content of mercury in fly ash. Then other metals were determined by the spectral analysis. By this approach, it is possible to determine the content of mercury in the whole volume of ash particles. The determination of the surface concentration of mercury was carried out by the XRF method (Olympus Delta X, model DPO, Innov-X Systems, Inc., USA) [18].

The concentration of mercury C_{Hg}^{P} could be expressed as a function of mercury mass m_{Hg} on the surface of ash particles:

$$C_{\mathrm{Hg}}^{P} = \frac{m_{\mathrm{Hg}}}{S_p} \times \frac{S_p}{m_{\mathrm{ash}}} \tag{1}$$

where: C_{Hg}^{P} – concentration of mercury $[g_{\mathrm{Hg}}/g_{\mathrm{ash}}]$ (recalculated as [mg/kg]), m_{Hg} – mercury mass, g, m_{ash} – fly ash mass, g, S_p – specific surface area of ash particles, m²/g.

The ratio of the surface area of particles and their mass is proportional to the size of particles. Thus Eq. (1) could be rewritten as:

$$C_{\mathrm{Hg}}^{P} = \frac{\beta}{D_p} \times \frac{m_{\mathrm{Hg}}}{S_p} \tag{2}$$

where: D_p – diameter of a particle, m, β – a constant.

From the dimensional analysis of Eq. (2) results that β is a reciprocal of the specific mass of ash. Equation (2) could be rewritten in the form:

$$\vartheta_{\mathrm{Hg}} = C_{\mathrm{Hg}}^{P} D_p \rho_s \tag{3}$$

where: ϑ_{Hg} – surface concentration of mercury, mg/m², ρ_s – density of solid particles, kg/m³. In order to verify Eq. (3), the XRF method was used to measure the mercury surface concentration for various types of fly ash obtained from the combustion of bituminous and lignite coals. The surface concentration of mercury has been studied on fly ash from individual ESP chambers.

Leaching of ash. The extraction (leaching) method allows one to separate mercury compounds. Water-soluble Hg in fly ash was determined by the batch leaching test. The liquid-to-solid ratio (L/S) was 10 and the release of Hg was measured for 24 h upon contact with distilled water. The aim of this experiment was to give an answer to the question if mercury adsorbed on the surface of ash particles is present mostly in an insoluble form (Hg^0 or HgO) or in the form of soluble salts form (first of all $HgCl_2$).

3. RESULTS AND DISCUSSION

3.1. ANALYSIS OF LIGNITE AND BITUMINOUS COAL ASHES

Analyzed ash has been separated on ESPs located in lignite and bituminous coal power plants. The average compositions of both types of coal under examination are given in Table 2.

Composition of investigated fossil fuels

Type of coal	C	H	N	S	Ash	Water	Hg	Cl	F
	[wt. %]						[mg/kg]		
Lignite	68.18	5.37	0.69	1.89	23.87	28.10	0.295	104.50	172.30
Bituminous coal	75.79	5.21	1.02	0.82	17.16	8.30	0.245	9850	166

Contents of water in raw fuel, of the remaining elements – in dry fuel.

In order to analyze the interaction between mercury and fly ash, the chemical composition of bituminous and lignite ash has been examined. Flying ash was dissolved in aqua regia and analyzed by the AAS method. The results are presented in Table 3.

T a b l e 3

Composition of ash from bituminous and lignite coals

Species	Bituminous coal	Lignite	Species	Bituminous	Lignite
SiO_2, wt. %	50.8	51.4	TiO_2, wt. %	1.10	1.30
Al_2O_3, wt. %	24.9	4.81	MnO, wt. %	0.101	0.024
CaO, wt. %	3.86	0.864	Cu, mg/kg	95	46
MgO, wt. %	2.20	0.301	Ni, mg/kg	95	78
Fe_2O_3, wt. %	6.87	14.7	Pb, mg/kg	119	12
Na_2O, wt. %	0.755	0.266	Hg, mg/kg	0.345	0.410
K_2O, wt. %	3.13	0.252	Loss of ignition	2.93	1.98

The composition of ash obtained from bituminous coal and lignite differs significantly, especially in the content of potassium, lead and magnesium. Ash from bituminous coal contains large quantities of oxides of silica, aluminum and iron, which also confirm, e.g., Wilcox et al. [10]. On the other hand, lignite ash contains larger quantities of CaO. Some oxides such as those of iron, copper and lead are enhancing oxidation and thus could also have catalytic effects on oxidation of HCl to Cl_2. The formed chlorine could react with vapors of mercury to mercury dichloride.

3.2. ASH PARTICLES SIZE AND SHAPE ANALYSIS

The SEM tests revealed that the ash particles are nearly spherical and it is obvious, according to Fig. 2, that they are heterogeneous, some of them porous when others nearly smooth. Very small particles are attached to the surface of larger ones. The formation of agglomerates confirms that the surface of large particles has been exposed to melting and small ash particles were then attracted. With regard to the size and shape of ash particles, it is highly probable that mercury would be adsorbed in small quantities

on the ash surface. It is worth mentioning that the particle size distribution is also changing with time.
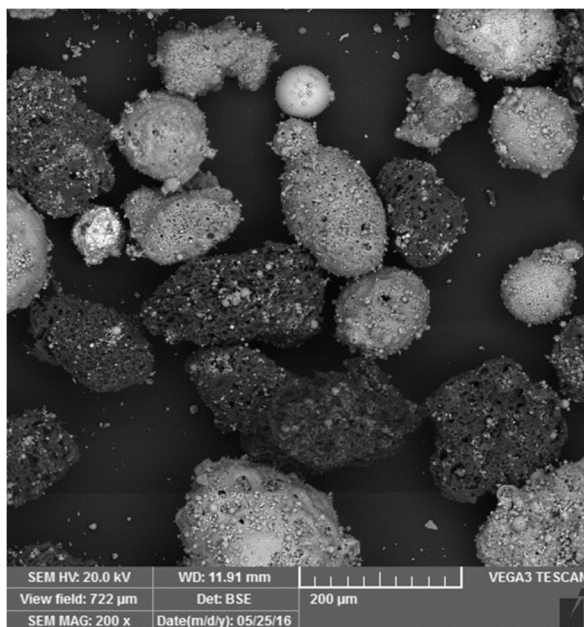

Fig. 2. Scan of lignite ash. Dark porous particles contain higher quantity
of carbon having the size and shape similar to the ash particles
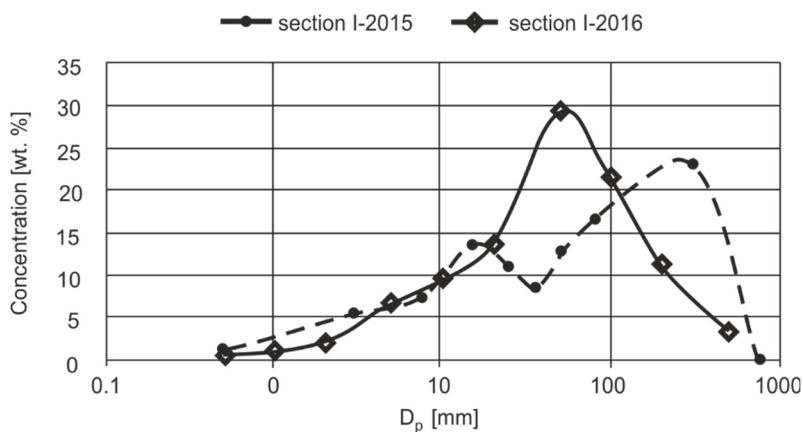

Fig. 3. Change in distribution of ash particle size in the first ESP chamber
in lignite combusted power unit within the period of 6 months

In Figure 3, the particle size distributions D_p of fly ash in the first ESP chamber for lignite combusted power unit are been presented within the six-month interval. The

average size of ash particles in 2015 (initial state) was 81 µm and after 6 months the average size has changed to 96 µm. It is worth mentioning that there also existed particles of the diameter larger than 500 µm for which the terminal particle velocity was 0.09 m/s.
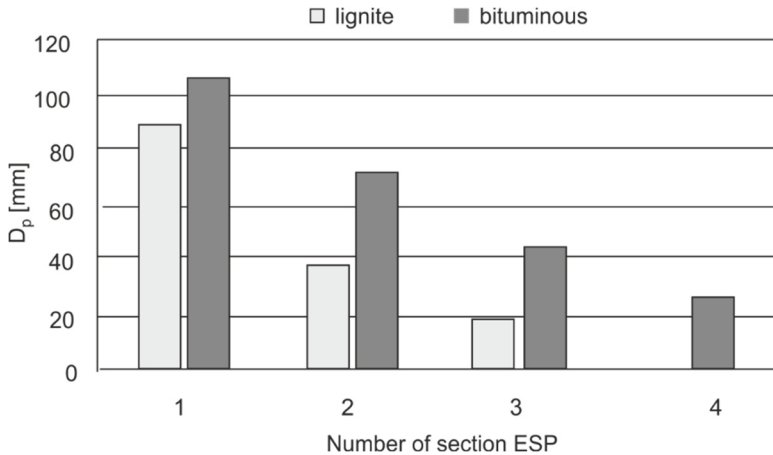

Fig. 4. Size of ash particles of brown coal (lignite) and bituminous coal
in individual chambers of ESP

Characteristic ash particle sizes in individual chambers for bituminous and lignite coal are shown in Fig. 4. An important property of separated fly ash from ESP is its ability to pour freely. Ash is usually transported pneumatically and then deposited. For these reasons, it is important that the particles do not agglomerate. Hence ESP is operated at temperatures higher than the dew point of exhaust gases because at this situation moisture does not support agglomeration process. The temperature in ESP during measurements was 150–200 °C.

3.3. ANALYSIS OF MERCURY CONCENTRATION ON ASH PARTICLES

The results of fly ash analysis from lignite and bituminous coals are summarized in Table 4. Experimental and calculated surface concentrations of mercury on bituminous coal and lignite ash separated in ESP are given in Fig. 5. The experimental results show that concentrations of mercury on ash determined by the XRF method and by the total decomposition method are similar. For this reason, it has been concluded that mercury is present only on the surface of the ash. This means that during the combustion of coal in the furnace particles of ash are firstly formed and next transported to ESP unit. On this distance, due to the change of ash temperature, mercury condenses on the surface of ash particles. Its quantity depends on initial concentration of mercury in exhaust gases and operating temperature inside ESP unit.

The results of fly ash analysis from lignite and bituminous coals taken from ESP chambers
of selected electric power plants (PPI or PPII) in the Czech Republic

| Coal/power plant /chamber	C_{Hg}^P [mg/kg]	ρ_s [kg/m³]	D_p [µm]	ϑ_{Hg} [µg/m²]	ϑ_{Cu} [µg/m²]	ϑ_{Pb} [µg/m²]	ϑ_{Cu+Pb} [µg/m²]	Loss of ignition [wt. %]
Lignite/I/1	0.151	1102	102	21.1	10	42.5	52.5	3.4
Lignite/I/2	0.385	1102	50	19.3	22.7	35.4	58.1	4.1
Lignite/I/3	0.392	1102	22.4	11.7	3.8	8.4	12.2	2.7
Lignite/I/3	0.437	1102	24	15.1	2.6	5.1	7.7	2.6
Lignite/I/1	0.187	1102	81.8	14.8	6.9	38.8	45.7	3.3
Lignite/I/2	0.465	1102	18.5	11.3	12.2	26.4	38.6	1.9
Lignite/I/3	0.625	1102	9.2	9.8	8.6	10.2	18.8	2.7
Lignite/II/2	0.436	1102	38.4	20.9	21.1	52.6	73.7	3.4
Lignite/II/2	0.487	1146	32.4	17.2	18	58.3	76.3	1.6
Lignite/II/1	0.291	1146	62.8	26.4	28.8	81.2	110	4
Lignite/II/1	0.237	1146	85.1	30.1	31	66.7	97.7	2.4
Lignite/II/3	0.611	1146	16.5	19.4	28	18.6	46.6	3.7
Bituminous/–/1	0.273	1292	105.6	42.8	68.3	125.4	193.7	1.07
Bituminous/–/2	0.574	1292	71.3	50.7	88.3	162.7	251	2.73
Bituminous/–/3	0.826	1292	44	47.1	89.8	121.3	211.1	4.1
Bituminous/–/4	1.05	1292	25.5	33	71.1	115.2	186.3	4.04
Bituminous/–/2	0.391	1292	90	41.3	86.2	145.2	231.4	3.02
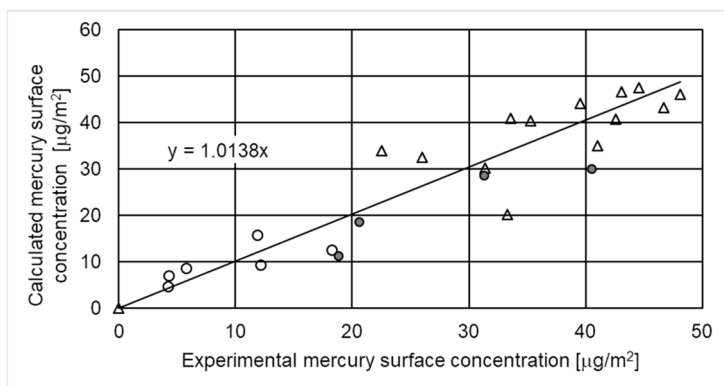
Fig. 5. Experimental and calculated surface concentrations of mercury on fly ash from bituminous coal
and lignite of power plant I and II: ▲ bituminous coal, ○ lignite PPI, ◑ lignite PPII. Verification of Eq. (3)

Ash from ESP is stored in an ash disposal area. For these reasons, it is important to
analyze in which form mercury occurs. The main criterion is capability of mercury (and
especially of mercury compounds) to be extracted (leached). From this point of view
only HgS is considered to be a harmless waste. Reaction conditions inside pipelines
enable reaction of mercury atoms with components present in the exhaust gases. The

type of formed compounds depends on the temperature and stability in the given location. At temperatures typical of ESP, first of all, HgO, $HgCl_2$, $HgSO_4$ and obviously Hg are present.

<div align="right">T a b l e 5</div>

Mercury concentrations in fly ash determined by the leaching method

Coal/power plant /chamber	Mercury on fly ash from ESP		Leachate from fly ash		
	Before leaching [mg/kg]	After leaching [mg/kg]	Chlorides [mg/dm^3]	Mercury [µg/dm^3]	pH
Lignite/I/1	0.202±20%	0.185±20%	<1	<0.02	6.2
Lignite/I/2	0.156±20%	0.146±20%	<1	<0.02	5.6
Lignite/I/3	0.407±20%	0.363±20%	<1	<0.02	6.1
Lignite/I/2	0.360±20%	0.424±20%	8.05±15%	<0.02	5.8
Lignite/I/3	0.427±20%	0.447±20%	10.8±15%	<0.02	6.5
Bituminous/–/1	0.367±20%	0.394±20%	12.4±15%	0.032±10%	5.8
Bituminous/–/2	0.764±20%	0.721±20%	14.7±15%	0.044±10%	6.1

The concentrations of mercury in fly ash are given in Table 5. Above analysis shows that only 2% of the mercury was obtained by leaching bituminous coal ash regardless of the size of the particles. On the contrary, mercury was not obtained on the fly ash from lignite. Higher content of chlorine in exhaust gases could activate atomic mercury and change it to soluble chloride.

3.4. CORRELATION BETWEEN MERCURY CONCENTRATION AND ASH PARTICLE SIZE

Individual ESP chambers separate fly ash particles after combustion of lignite and bituminous coal which differ one from another, first of all, by the size of particles. In Fig. 6, mercury concentrations are plotted as functions of the size of ash particles.

It could be concluded from Fig. 6, that there exist particles of fly ash both from bituminous coals and lignite where the quantity of mercury adsorbed on the ash surface is the highest. Using the least square method, the polynomials of second degree were fitted to the data. For both types of ash, the maxima of the fitted parabolic functions lie in the neighborhood of $D_p = 67$ µm, although the achieved maximum concentrations of Hg are different. It is worth mentioning that presented analyses were made both for lignite and bituminous coal obtained from Krušné mountains and Silesian field, respectively. As can be seen in Fig. 7, the specific concentration of mercury on the surface of ash was proportional to the size of ash particles. It was also observed that there existed ash particles with the diameter in the range from 50–60 µm that exhibited maximum capability to adsorb on their surface mercury and its compounds. The results of examinations reported by other authors confirm these observations [19, 20].
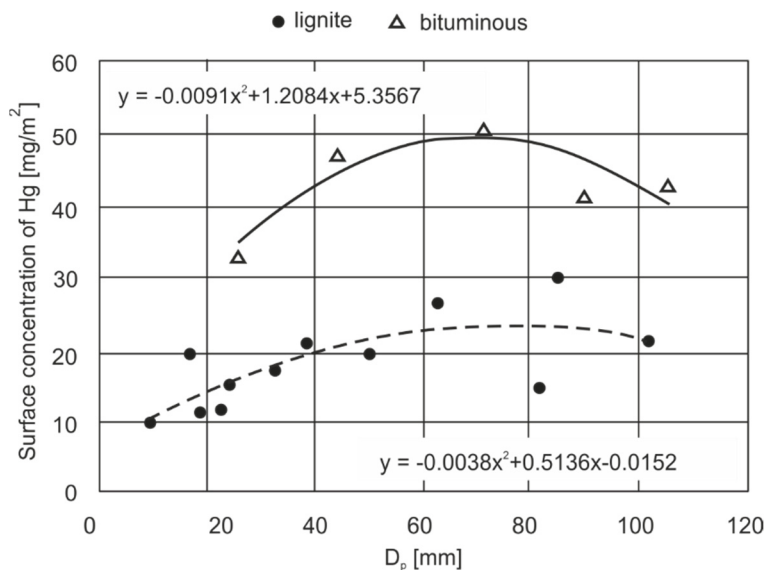
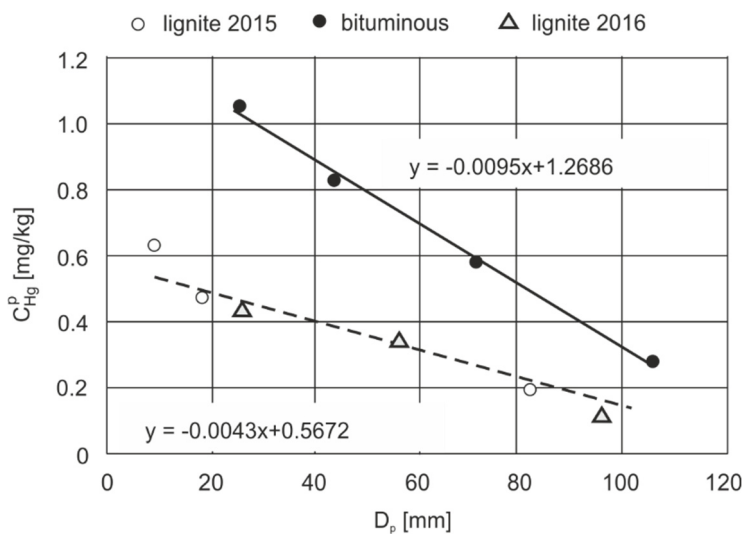Fig. 6. Experimental values of mercury concentrations depending on the size of ash particles

Fig. 7. Dependence of mercury concentration on average ash particle size obtained
from lignite and bituminous coal

The greatest effect on Hg concentration had the surface concentration of copper and lead (Fig. 8). Both these metals form amalgams with mercury. It is probable that copper and lead form active centers on the ash surface preferably occupied by molecules

containing mercury atom. It is thus possible to expect that content of these elements in fly ash may have a significant effect on sorption of mercury from exhaust gases.
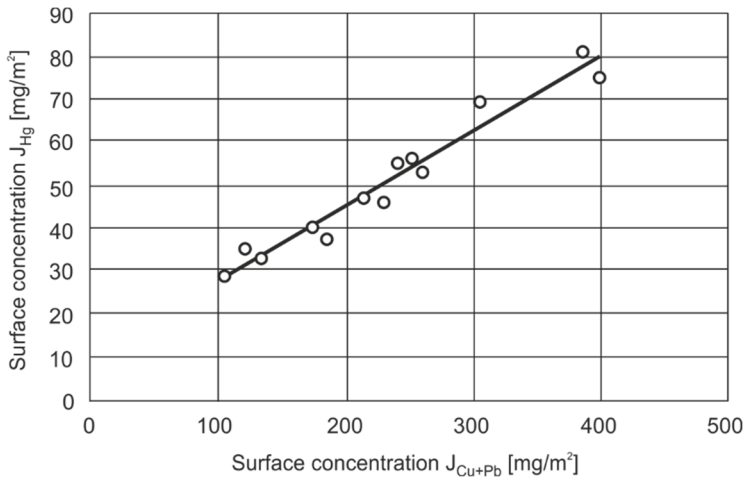
Fig. 8. Effect of copper and lead presence on adsorption of mercury on ash surface

The dependence of adsorbed mercury on fly ash from bituminous coal and lignite on the content of unburned coal was studied. The concentrations of unburned coal in all samples of fly ashes (from bituminous coal and lignite) was at the maximum level up to 4% of its mass. The obtained results could not have been correlated. It was thus possible to assume that the quantity of unburned carbon in ash would depend on the combustion regime and that its effect on sorption of mercury could not be clearly proved.

4. CONCLUSIONS

Ash particles, separated from ESPs, are a natural sorbent. Mercury and its compounds condense on the ash surface and are adsorbed. Experimental results showed that concentrations of mercury on fly ash determined by the XRF method and by total decomposition give the same values. For these reasons it has been concluded that mercury is present only on the surface of ash particles. It was also found that mercury concentration on ash surface increases upon diminishing size of ash particles. The ash particles of the diameter from 50 to 60 μm exhibit maximum capability to adsorb mercury and its compounds. Moreover, lead and copper ions form active centers on ash surface that contribute to increase of sorption capacity of mercury and of its compounds. It is highly possible to expect that content of these elements in fly ash would have a significant effect on sorption of mercury from exhaust gases.

ACKNOWLEDGEMENTS

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