Chloride and Fluoride Contents in Flue Gas During Domestic Lignite Coals Combustion as a Parameter in the Design of Flue Gas Desulphurisation Plant

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Recently, research in the field of coal combustion include impurities, specifically halogen elements (F, Cl, Br, I and At). Emission of chlorides and fluorides from the combustion depends on content and forms of these elements in coal, combustion process and emission reduction equipment. Examination of chlorides and fluorides content in coal and in flue gas is particularly important for design of flue gas desulphurisation plant, the integral part of the modern power plants which ensure meeting the requirements of SO_2 emission regulations. In flue gas desulphurisation facilities, the presence of HCl may increase sorbent consumption and HCl and HF have the influence on wastewater treatment. This paper presents the results of chlorine and fluorine contents in domestic lignites and their concentration in flue gas. The aim of investigation was to determine the reference Cl and F concentrations in flue gas that would be used in the design of flue gas desulphurization plant.

Keywords: *power plants, chlorine, fluorine, flue gas, desulphurisation,*

1. INTRODUCTION

Steady rise in world electricity consumption imposes the development of new and improvement of existing coal combustion technologies in large power plants as well as installations and other equipment for flue gas treatment to meet increasingly strict environmental regulations. In the past years, research of the coal combustion process was devoted, among other things, to studying the content and transformation of halogen elements (F, Cl, Br, I and At). According to reactivity, halogens can be classified as follows F>Cl>Br>I [1], hence most of the research is related to Cl and F. During combustion the halogens are transferred to gas and under favourable conditions form acids, causing problems in both power plants' installations and their environment. Chlorides have corrosive effects on evaporator and super heater tubes, but also emerge in the flue gas desulphurization process [2, 3, 4]. If chlorides escape into the atmosphere, they cause aerosol occurrence [5]. Fluorides are very toxic and in high amounts may be hazardous for humans and the living world [6].

Chloride and fluoride emissions from combustion depend on the content and form of these elements in coal, combustion conditions and characteristics of installations for emissions reduction. Differences in fluoride and chlorine contents are not only a consequence of the original matter structure and coal transformation in the process of its formation (because chloride and fluoride occurrence is not determined by coal rank only) but also emerge inside the same coal basin.

Chlorine in coal comes from the original matter that coal was formed from and research established that there are three main forms of chlorine in coal [7, 8]:

- chlorine ions in saltwater and other water-bound compounds in coal – NaCl, KCl, $CaCl_2$ and sometimes $MgCl_2$ and $FeCl_2$;
- organic compounds-bound chlorine – organic (is present bonded in organic macromolecules), and
- inorganic chlorine.

Recent studies indicate that chlorine is mostly bonded in the first of above mentioned states and that in the form of crystal water-bound chlorine anion in coal pores. In drying process, chlorine precipitates to give NaCl [9, 10].

The most frequent form of fluorine inorganic occurrence is mineral fluorapatite ($Ca_5/(PO_4)_3$ (F, OH, Cl)). Other modes of occurrence of fluorine include fluorite CaF_2, biotite/mica $K(Mg, Fe)_{2\text{-}3}Al_{1\text{-}2}Si_{2\text{-}3}O_{10}(OH,F)_2$, amphiboles (a group of ferromagnesian silicate minerals), and in tourmaline and topaz [11]. In the structure of clay minerals the bonding of fluorine ions F— is possible instead of OH—[12]. In the literature, in the past couple of years, the existance of organically-bound fluorine was proved [13, 14].

Chlorine content in coal varies from several ppm to several thousand ppm, and for the majority of coals it is within range of 50-2000 ppm, although it can be much higher in coals from some basins. Yudovich and Ketris determined the mean concentrations of chlorine (at

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global level) from 120±20 ppm in lignites and sub-bituminous coals, and from 340±40 ppm in anthracites and bituminous coals. Differences in Cl concentrations in coal between coal basins can be pronounced and can deviate considerably from mean value. Also, differences in Cl concentrations in coal basin have been observed to be depth – related. Cl concentration increases with increase of depth, due to water presence [7]. Concentration of Cl compounds rises as high as ~86% of coal C content [8].

Mean fluorine concentration is 90±7 ppm for lignites and sub-bituminous coals, and 82±6 ppm for anthracites and bituminous coals. Concentrations in ash are 630±50 ppm and 580±20 ppm, respectively [16]. Concentrations increase with increase of depth, decreasing after a certain depth, when a maximum value is reached. As a rule of a thumb, mean fluorine concentration rise with coal rank [12]. Depending on the method employed to determine fluorine content, it can vary largely in different types of coal, hence, in some cases it is difficult to compare coal fluorine content and its properties during combustion processes reported in various studies. Swaine [5] found that the fluorine content in coals is in the range 20-500 ppm with mean value of approx. 150 ppm.

A wide range of methods have been developed for determining chlorine and fluorine content in coal which can be classified into two groups: quantitative (standard and non-standard) and extraction methods. Studies of chlorine and fluorine content indicate differences in results obtained by applying different methods or from different laboratories, which are explained by different forms of coal mass-bound chlorine and fluorine.

Chlorine emissions from coal-fired plants range from 50 to several thousand ppm, depending on chlorine content in coal, type of boiler and installed equipment for pollutants control. Water-soluble chlorine, which is present in coal and bound to coal structure by weak bonds, is very quickly transferred into a gas phase during pulverized coal combustion. The remaining amount of organic mass bound chlorine in coal is released during combustion of carbon. In practical terms, the whole amount of vaporized chlorine occurs in combustion products in the form of HCl. Elemental chlorine may occur during the oxidation process in the presence of metal oxides that behave as catalyst in fly ash and boiler deposits. Thus HCl leaving the chimney is in vapor state, although a certain amount of HCl may be adsorbed in smaller fly ash particles. Fluorine emissions are generally lower compared to chlorine emissions, because fluorine content in coal is lower. The largest amount of fluorine in coal passes to HF during the combustion process, while only a smaller amount (< 10%) may remain in slag. Also, a smaller amount of HF may be adsorbed in fly ash particles prior to their leaving the stack. According to investigations [17], an assumption was introduced that fluorine occurs in two different forms that behave differently during the combustion process. The first form accounting for 25 – 50 % of total fluorine is water – soluble emitted as gaseous HF, and the second one (50-75 %) is water insoluble and inert during the combustion process and remains in ashes.

In flue gas desulphurization plants HCl from flue gases is absorbed faster than sulphur dioxide, so the presence of HCl may increase requirements for sorbent. In the pre-absorber a larger amount of fly ash and dissolved gases is separated, such as HCl and HF, and wastewater is discharged to a water treatment plant. The wastewater treatment is determined by HCl and HF contents in dissolved gases. High concentration level of halogens, particularly of chlorine (Cl), in suspension inside the absorber causes metal point corrosion. Ions of the halogens penetrate easily through protective passive film, particularly in points of micro cracks or material physical and chemical heterogeneity. Also, fluorine reacts with aluminum oxide to form ion AlF_6^{3-} causing deposits inside the absorber difficult to remove. Because of these reasons chlorine and fluorine concentrations are always the subject of studies in flue gas desulphurization plant design aiming to keep their concentration below allowable levels [18].

This paper presents the results of analyses of chlorine and fluorine contents in domestic lignites and their concentration in flue gas developed at pulverized coal combustion in thermal power plants. The aim of studying the analyses results was to determine the reference Cl and F concentrations in flue gas that would be used in the design of flue gas desulphurization plant.

2. EXPERIMENTAL TESTS

Experimental analyses of chlorine and fluorine contents in coal and their transformations during the combustion process were carried out at the units of Thermal Power Plant Nikola Tesla (TPPNT), which use low-calorific coal - lignite from Kolubara open pit mines.

Studies were conducted under real conditions and at common operating modes of coal fired units that use coal from different mining pits of Kolubara coal basin: Tamnava, Vreoci Stari and Vreoci Novi, as well as coal from power plants' coal depot. Studies were performed at the TPPNT units B1 (620 MW of power capacity, symbol B) and at the TPPNT units A6 (350 MW of power capacity, symbol A). Four studies were carried out for each unit (symbols I-IV). Each study included two series of analyses, 3 hours each at a time, so the total duration of each experiment was 6 hours.

Coal sampling was performed from a coal feeder every 30 minutes and finally a composite sample was formed for each experiment. At units with two flue gas ducts (right and left), simultaneous measurements were performed of hydrogen chloride and hydrogen fluoride concentrations. Along with sampling performance and its dynamics, unit operating parameters and flue gas characteristics were recorded.

To obtain representative results, two different methods for determining chlorine and fluorine contents in coal were employed. Chlorine content in coal was determined using the ISO 587 [19, 20] and ASTM D4208 [21] methods, while the ASTM D5987 [19, 20] and ASTM D3761 [21] methods were applied to determine fluorine content in coal.

Hydrogen chloride and hydrogen fluoride concentrations in flue gas were determined by the EPA Test method 320: 1999. This method was also employed to determine oxygen and moisture contents in flue gas.

3. TEST RESULTS

Results include coal characteristics exhibited during experiments (Tables 1 and 2) and unit operating parameters as well as flue gas characteristics (Tables 3 and 4).

Table 1. Ultimate and proximate analysis of coals used during studies at TPPNT B1 (as received mass) [19]

Series of experiments	I-B	II-B	III-B	IV-B
Coal	Tamnava	Tamnava	Tamnava/ Vreoci Novi	Tamnava/ Vreoci Stari
W (*%m/m*)	49.90	49.30	49.50	48.80
A (*%m/m*)	14.51	16.94	19.16	23.21
C (*%m/m*)	23.15	21.74	20.24	18.18
H (*%m/m*)	1.98	1.93	1.81	1.69
O+N (*%m/m*)	10.22	9.74	8.91	7.69
S (*%m/m*)	0.24	0.36	0.38	0.43
H_d (kJ/kg)	7819	7245	6639	5811

Table 2. Ultimate and proximate analysis of coals during studies at TPPNT A6 (as received mass) [20]

Series of experiments	I-A	II-A	III-A	IV-A
Coal	Tamnava*/ Vreoci Novi (2/3) and depot coal (1/3)**	Vreoci Novi (80%) and mixture of depot coal and Tamnava (20%)*/ Vreoci Novi (80%) and depot coal (20%)**	Tamnava*/ Vreoci Stari (30%) and depot coal (70%)**	Vreoci Stari (1/3) and Tamnava (2/3)*/ Vreoci Stari (1/3) and Tamnava (2/3)**
W (*%m/m*)	49.40	48.00	50.60	49.70
A (*%m/m*)	19.84	18.15	17.69	17.38
C (*%m/m*)	19.33	21.87	20.25	21.46
H (*%m/m*)	1.78	1.96	1.85	1.91
O+N (*%m/m*)	9.13	9.58	9.32	9.15
S (*%m/m*)	0.52	0.44	0.28	0.41
H_d (kJ/kg)	6272	7320	6623	7128

* first (morning) series

** second (afternoon) series

Table 3. Mean values of operating parameters for the TPPNT B1 unit during experiments [22]

Series of experiments	I-B	II-B	III-B	IV-B
Coal	Tamnava	Tamnava	Tamnava*/ Vreoci Novi**	Tamnava*/ Vreoci Stari**
Unit power capacity [MW]	617	599	590	603
Moisture content in flue gas [%]	22.3	20.6	23.2	23.1
O_2 content in flue gas [%]	4.9	5.1	4.8	5.1
Flue gas temperature [°C]	169	177	180	178
Flue gas flow [m^3/h]***	2,641,023	3,187,800	3,003,808	3,039,844

* first (morning) series

** second (afternoon) series

*** dry flue gas @ NPT (101,3 kPa, 0 °C), ref. O_2 content (6 *%v/v*)

Comparative results for determining chlorine and fluorine contents in coal by applying different methods are shown in Figs 1 and 2 [19, 20, 21]. Results for measurements of the chlorides and fluorides content in flue gas [22] are presented in Figs 3 and 4. The contents of chlorides and fluorides in flue gas (calculated on a dry flue gas basis, 6 %v/v O2, NPT) for the whole period of experiment (two 3-hour series each) are given in Fig. 5.

Table 4. Mean values of operating parameters for the TPPNT A6 unit during studies[22]

Series of experiments	I-A	II-A	III-A	IV-A
Coal	Tamnava*/ Vreoci Novi (2/3) and depot coal (1/3)**	Vreoci Novi (80%) and mixture of depot coal and Tamnava (20%)*/ Vreoci Novi (80%) and depot coal (20%)**	Tamnava*/ Vreoci Stari (30%) and depot coal (70%)**	Vreoci Stari (1/3) and Tamnava (2/3)*/ Vreoci Stari (1/3) and Tamnava (2/3)**
Unit power capacity [MW]	328	344	330	289
Moisture content in flue gas [%]	22.9	22.1	19.1	19.1
O_2 content in flue gas [%]	5.7	5.5	5.6	5.8
Flue gas temperature [°C]	164	157	161	161
Flue gas flow [m^3/h]***	1,376,100	1,476,000	1,468,800	1,423,800

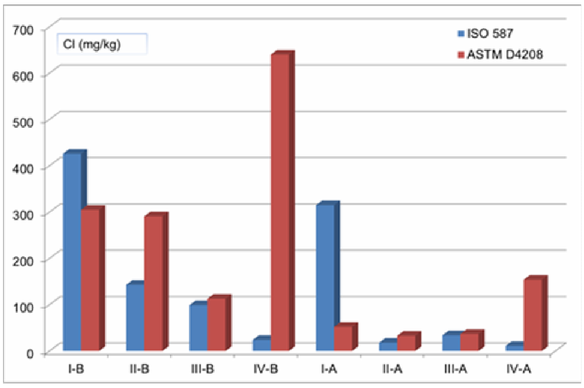

Figure1. Chlorine content in coal determined by different study methods (calculated on dry mass basis)

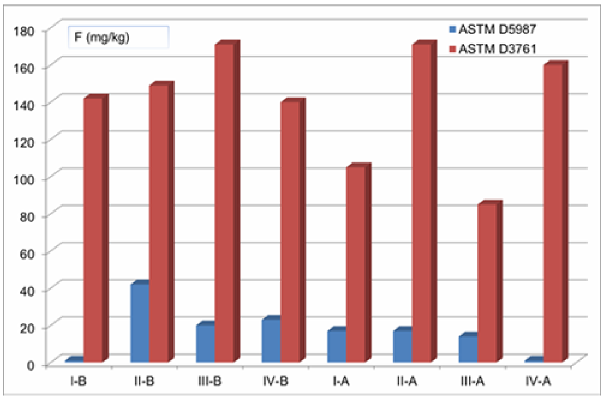

Figure 2. Fluorine content in coal determined by different study methods (calculated on dry mass basis)

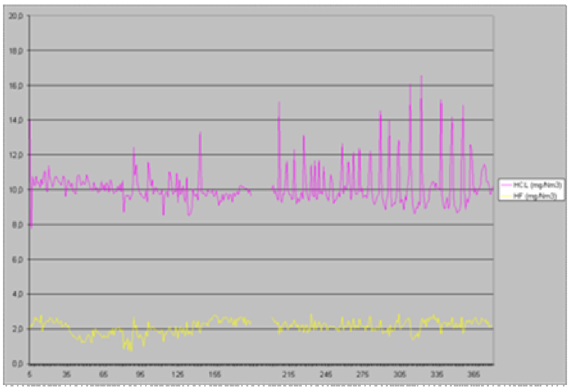

Figure 3. Values of HCl and HF contents measured in flue gas at TPPNT B1, during measurements at I-B (real flue gas concentrations)

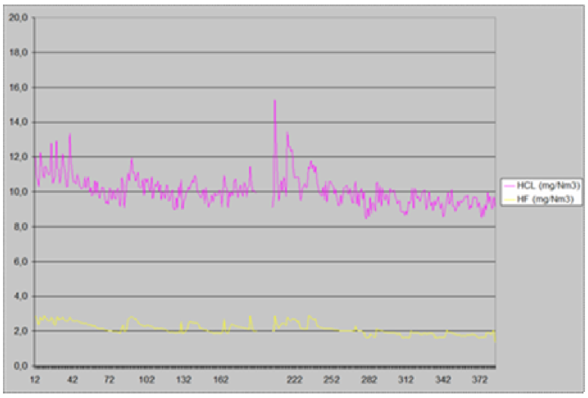

Figure 4. Values of HCl and HF contents measured in flue gas at TPPNT A6, during measurements at I-A (real flue gas concentrations)

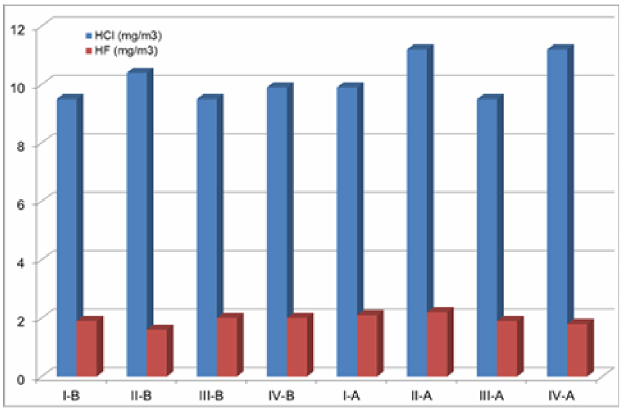

Figure 5. Contents of hydrogen chloride and hydrogen fluoride in flue gas per series of experiments (dry gas, 6 %*v/v* O_2, NPT)

4. DISCUSSION

Based on analyses of the chlorine and fluorine contents in coal determined by different standard methods, significant differences are noted between the results obtained (Figs 1 and 2). Within the framework of analyzed samples, chlorine concentration was in the range 24÷426 mg/kg (according to ISO 587 method) and 113÷640 mg/kg (according to the ASTM D4208 method), respectively. Mean concentrations were 134 mg/kg and 203 mg/kg, respectively. Comparing these concentrations with chlorine mean concentrations at global level, it is noted that obtained results for the coals from Kolubara coal basin correspond, according to the ISO 587 method, to the values complying with literature data on lignites, whereas the ASTM D4208 method produces results corresponding to a group of coals between lignite and sub-bituminous coals, on one hand, and to bituminous coals and anthracite, on the other hand.

Within the framework of analyzed coal samples, fluorine concentration ranged from 1÷42 mg/kg (according to the ASTM D5987 method) and from 85÷171 mg/kg (according to the ASTM D3761 method), respectively. Mean concentrations are 17 mg/kg and 140 mg/kg, respectively. Comparing these concentrations with mean fluorine concentrations in lignites and sub-bituminous coals, at global level, the ASTM D3761 method produced values twice as much as mean values for fluorine concentration reported in literature. Mean values of fluorine concentration in coal, obtained by averaging samples according to the ASTM D5987 method is much lower than those obtained by Yudovich and Ketris for means related to concentrations in bituminous coals and anthracite. For all experiments, chlorine concentrations in coal are higher than fluorine concentrations, which is in agreement with literature data [15, 16]. At the same time, the obtained values of chlorine and fluorine contents in coal agree with the values obtained in the study of coal samples from different coal pit mines in Kolubara coal basin [23, 24].

For TPPNT B1, hydrogen chloride concentration is within the 9.5 – 11.2 mg/m^3 range (calculated on a dry flue gas basis and 6 %*v/v* O_2), mean value being 9.8 mg/m^3. The coefficient of deviation is 4.3 %. Hydrogen fluoride concentration is in the 1.6-2.0 mg/m^3 range (calculated on a dry flue gas basis and 6 %*v/v* O_2), while mean value is 1.9 mg/m^3. The coefficient of deviation is 10.1 %. For TPPNT A6, hydrogen chloride concentration is within the range of 9.5-10.4 mg/m^3 (calculated on a dry flue gas basis and 6 %*v/v* O_2), mean value being 10.5 mg/m^3. The coefficient of deviation value equals 4.8%. Hydrogen fluoride concentration is in the 1.8-2.2 mg/m^3 range (calculated on a dry flue gas basis and 6 %*v/v* O_2), where mean value amounts to 2.0 mg/m^3. The coefficient of deviation is 9.1 %.

In flue gas, hydrogen chloride concentrations are higher than hydrogen fluoride concentrations. There are slight differences between hydrogen chloride and hydrogen fluoride mean values when studied at different units (<10 %).

Using data on measured chlorine and fluorine content, maximum concentrations of chlorides and fluorides in flue gas were determined, assuming that the total amount from coal was transformed into gases in the flue gas (Figs 6 and 7). The calculated HCl concentrations for the IV-B series of study (according to the result for Cl by the ISO 587 method) are higher, except for one result, than those measured. In the TPPNT A6 series of experiments, the trend is that calculated values are lower than those measured (except for two results). For the series of fluorine content determination according to the ASTM D3761 method, the calculated HF concentrations in flue gas are substantially higher than those measured, and for the series of F content determination according to the

ASTM D5987 method it is not possible to deduce a single-valued conclusion.

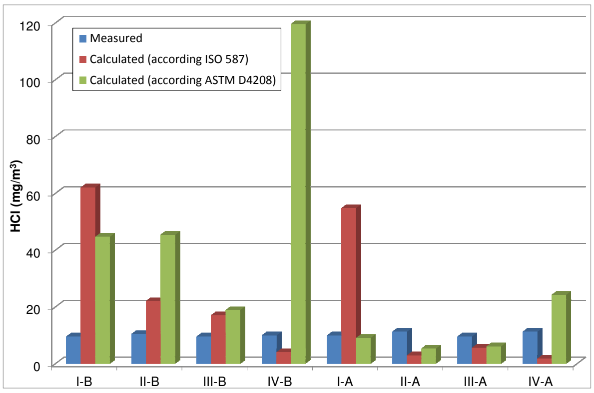

Figure 6. Comparative representation of obtained and calculated HCl concentrations per Series of experiments at TPPNT B1 (dry gas, 6% v/v O_2, NPT)

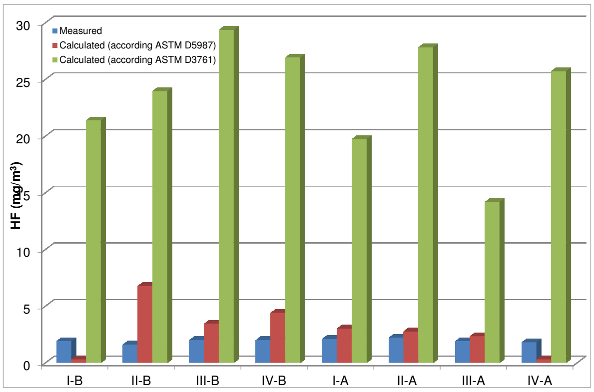

Figure 7. Comparative representation of obtained and calculated HF concentrations per Series of experiments at TPPNT B1 (dry gas, 6% v/v O_2, NPT)

5. CONCLUSIONS

Based on performed experiments on determining of chloride and fluoride contents in flue gas for the design of flue gas desulphurization plant, the following can be concluded:

– applied standard methods indicate significant differences in Cl and F contents in coal, that is consequence of analyzing blend of coals from different open pit mines of Kolubara coal basin, taken directly from a coal feeder,

– methods used to determine Cl and F content in coal produce significant differences when the same coal samples are analyzed,

– obtained results in this study for Cl and F content in coal agree with the values of Cl and F contents in the Kolubara basin coals obtained by other researches for analyzed samples from the same open pit mines,

– Cl concentrations are higher than F concentrations. Maximum measured Cl and F concentrations in coal amounts to 640 mg/kg and 171 mg/kg, respectively, and these values should be taken into account in further considerations of the flue gas desulphurization process and equipment,

– the measured values of HCl and HF concentrations in flue gas are virtually constant during studies,

– maximum HCl and HF values (dry gas, 6 *%v/v* O_2, NPT), calculated for the total Cl and F conversion to HCl and HF, amounts to:

	TPPNT B1	TPPNT A6
HCl	116 mg/m^3	56 mg/m^3
HF	28 mg/m^3	27 mg/m^3

and these values are to be considered in the design of flue gas desulphurization equipment.

Bearing in mind above mentioned conclusions, primarily differences in measurement results for HCl and HF contents in flue gas and values obtained by calculations based on material balance, the requirements for future studies must include monitoring of Cl and F content in coal and fly ash along with simultaneous parallel measuring of HCl and HF in flue gas using the reference and FTIR method. Those studies would allow determination of the conversion factor i.e. determination of Cl and F binding from coal in fly ash.

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САДРЖАЈ ХЛОРИДА И ФЛУОРИДА У ДИМНОМ ГАСУ ТОКОМ САГОРЕВАЊА ДОМАЋИХ ЛИГНИТА КАО ПАРАМЕТАР ПРИ ПРОЈЕКТОВАЊУ ПОСТРОЈЕЊА ЗА ОДСУМПОРАВАЊЕ ДИМНОГ ГАСА

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Од недавно поље истраживања сагоревања угља обухвата и истраживање нечистоћа, посебно халогених елемената (F, Cl, I и At). Емисије хлорида и флуорида из процеса сагоревања зависе од садржаја и облика ових елемената у угљу, процеса сагоревања и опреме за смањење емисија. Испитивање садржаја хлорида и флуорида у угљу и

димном гасу је посебно значајно при пројектовању постројења за одсумпоравање димног гаса, саставног дела модерних термоелектрана, које обезбеђује испуњење прописаних захтева у погледу емисије SO_2. У оквиру система за одсумпоравање димног гаса, присуство HCl може довести до повећања потрошње сорбента, а HCl и HF имају утицај на прераду отпадних вода. У овом раду су приказани добијени резултати садржаја хлора и флуора у домаћим лигнитима и њихова концентрација у димном гасу. Циљ истраживања је било утврђивање референтних концентрација Cl и F у димном гасу које ће бити коришћене при пројектовању постројења за одсумпоравање димног гаса.