

<https://doi.org/10.3176/oil.1999.1.05>

REACTIVITY OF OIL SHALE ASHES TOWARDS SULFUR DIOXIDE

2. LOW-TEMPERATURE ASHES FORMED BY USING CFBC TECHNOLOGY

R. KUUSIK
T. KALJUVEE
A. TRIKKEL

Tallinn Technical University,
Department of Basic and Applied Chemistry
5, Ehitajate Rd, Tallinn 19086 Estonia

H. ARRO

Tallinn Technical University,
Department of Thermal Engineering
116, Kopli St, Tallinn 11712 Estonia

Samples of different ashes formed during combustion of Estonian oil shale in a circulated fluidised bed (CFB) facility (in Hans Ahlström Laboratory, Karhula) were analysed by different methods. Binding activity of these ashes towards SO₂, and the influence of temperature and the previous grinding of samples on the binding activity were studied with thermogravimetric equipment. The absence of sulfide sulfur in these ashes and their high SO₂ binding capacity were established. At 800 °C and after 10-minute contact, up to 37 mg of SO₂ per 100 mg of sample was bound. Previous grinding of the ashes increased this value by 1.8 times, almost to the level of the SO₂ binding ability of natural limestone.

The SO₂ binding ability of the ashes studied was approximately twice higher than that of cyclone ash formed by using the pulverized firing technology.

Introduction

Energy production in the Republic of Estonia is based on the burning of a low-quality local solid fuel - Estonian oil shale (EOS). This accounts for 67 % of the Estonian primary energy and 98 % of the electricity production in the SC *Eesti Energia* is based on the using of oil shale [1,2]. Thermal power plants operate on the combustion technology based on pulverised firing of oil shale at atmospheric pressure (PF). The high content of ash in the oil shale causes fouling and corrosion of heat transfer surfaces of boilers which leads to the situation that the combustors do not work under the optimum conditions. The power plant

efficiency is on the level of 28-30 %. Besides, the high temperature of combustion of pulverized oil shale (~1400 °C) leads to the decrease in the activity of ash. So, in spite of the high mole ratio Ca/S (approximately 9-10) in oil shale, only 70-80 % of the sulfur present in oil shale is bound [2].

The modernisation of boilers and the introduction of environmentally harmless burning technologies are important issues. The most attractive of these methods is the combustion of fuel in a circulating fluidized bed furnace under atmospheric pressure (ACFBC) [3] or in pressurised conditions (PCFBC) [4]. In 1979-1998, 275 CFBC units were designed and manufactured world-widely, of which 242 units, with an average capacity of 132.4 MW, were made by Foster Wheeler and LLB Lurgi companies to whom the leadership position in the development of CFBC technology belongs [5]. The positive results of the combustion of low quality fuels including EOS [6-8] have created a basis for the use of CFBC technology for EOS combustion [9, 10].

The ashes formed in the CFB combustors, compared to the ashes formed in PF conditions, might be more active in SO₂ binding. It could be caused, firstly, by a higher activity of free CaO formed at lower temperature and, secondly, by continuously renewing surface of the ash particles due to the friction of the particles. This was confirmed by using grinding for activating the high-temperature ashes [11]. Essential differences in the burning conditions, especially in the temperature regimes of PF and CFBC technologies, bring about differences in the chemical composition, physical properties, and reactivity of ashes. The latter parameters have also a direct relationship with the contamination of the atmosphere with SO₂, with the conditions of the following storing and utilisation of ashes. The environmentally safe conditions of storing of ashes depend mostly on the potential content of CaS in the ashes formed in CFBC conditions [12].

As the former publications contain no data on these relationships, the aim of the present investigation was to study the chemical composition and reactivity towards SO₂ of ashes formed during combustion of EOS in a circulating fluidized bed.

Experimental

Materials

The actual experiments of combustion of EOS (fuel particle sizes 0-15 and 0-25 mm) were carried out in the ~1 MW_{th} Ahlström Pyroflow CFB test facility (∅ 0.6 m) in Hans Ahlström Laboratory (Karhula, Finland) in September 1994 by using two different boiler feeding modes - 94 % (Mode-1) and 50 % (Mode-2) [8]. The technological parameters of the two modes are presented in Table 1.

The samples of the ashes formed during combustion of EOS - the so-called low-temperature ashes (LTA) - were gathered from the bottom of the furnace (LFA-I and LFA-II), the cyclone (LCA-I and LCA-II),

circulating ash), the precipitation (connecting) chamber (LPCA-I and LPCA-II) and the bag filter (LBFA-I and LBFA-II) (Fig. 1).

Methods

The experiments for studying the SO_2 binding efficiency were carried out with Q-Derivatograph (MOM, Hungary) in isothermal conditions described in [11].

The content of the main components and main characteristics of the studied samples determined by different methods (chemical, BET nitrogen dynamic adsorption, SEM, etc.) are presented in Table 2. The grain composition of LTA is presented in Table 3, distribution of free CaO and CO_2 in different fractions in Table 4 and Table 5, respectively, and the specific surface area for different fraction in Table 6. The content of different form of sulfur in LTA is presented in Table 7. For comparison, a sample of high-temperature cyclone ash (HTA, CA) formed at the Baltic Power Plant and limestone from Karinu deposit was used.

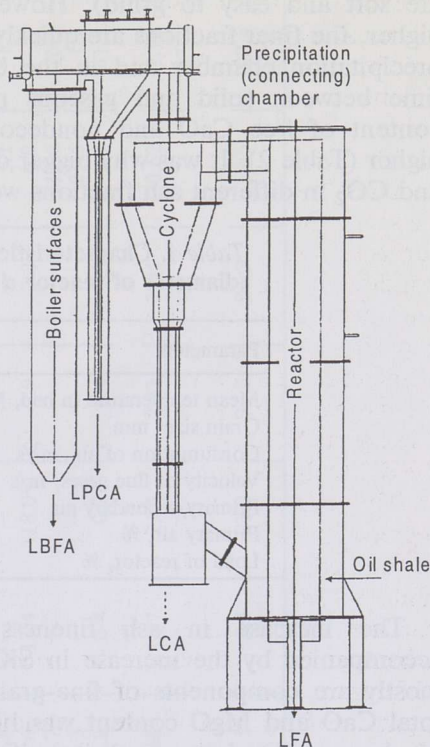


Fig. 1. The scheme of the 1 MW_{th} Ahlström Pyroflow pilot test facility

Results and Discussion

Characterisation of Ashes

The samples of furnace (LFA-1) and cyclone (LCA-1) ashes formed during fuel feeding Mode-1 are coarser and contain more free CaO and CO_2 than those of Mode-2. The samples of LPCA-1 and LBFA-1 are finer than the samples of LPCA-2 and LBFA-2, but the content of free CaO and CO_2 is, like in the previous case, higher than in the samples of Mode-2. The reason for this is that by using Mode-1 the load of the reactor and the velocity of the flue gases were approximately two times higher than by Mode-2 (Table 1). Consequently, friction between ash (fuel) particles is more intensive by using Mode-1 and the formation of finer fractional classes of ash would be more likely (Ca, Mg oxides formed in the combustion chamber during decomposition of carbonates

are soft and easy to grind). However, as the velocity of the gases is higher, the finer fractions are quickly carried away and precipitated in the precipitation chamber and in the bag filters (Table 3). As the contact time between solid and gaseous phases at Mode-1 was shorter, the content of free CaO and nondecomposed carbonates in ashes-1 were higher (Table 2). It was why bigger differences in the content of free CaO and CO₂ in different ash fractions were observed (Tables 4 and 5).

Table 1. Characteristics of Fuel Feeding Modes (diameter of reactor $d = 0.6$ m)

Parameters	Mode-1	Mode-2
Mean temperature in bed, °C	850	850
Grain size, mm	0-15	0-25
Consumption of air, m ³ /s	0.273	0.145
Velocity of flue gases, m/s	3.75	1.98
Primary/secondary air	130/200	90/85
Primary air, %	39	51
Load of reactor, %	94	50

The increase in ash fineness from LFA towards LBFA was accompanied by the increase in SiO₂, Al₂O₃ and Fe₂O₃ content, which mostly are components of fine-grain sandy-clay parts of oil shale. The total CaO and MgO content was higher in LFA and LCA (Table 2). A good correlation between the specific surface area and the content of free CaO in different fractional classes of ashes was observed (Table 6 and Table 4).

The higher content of SO₄²⁻ in LFA and LCA is caused, on the one hand, by the fact that the contact time of these ashes with the gaseous phase was much longer than that of LPCA and LBFA, and, on the other hand, by the higher content of free CaO in the furnace (bottom) ashes and the circulating ashes (Table 2).

One of the most serious problems when using the CFBC technique is related to the different forms of sulfur bound in the ashes and, hence, the storage of these ashes. Being water-soluble, the sulfide form of sulfur could be a dangerous source of contamination of the environment.

In oxidising conditions, the SO₂ binding in the ash takes place according to the following chemical reactions [12]:

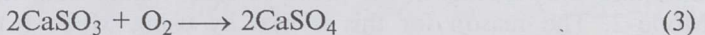
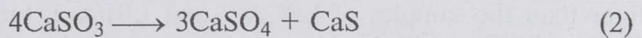


Table 3. Granulometric Composition of Ashes, %

Fractional class, mm	Sample							
	LFA-1	LCA-1	LPCA-1	LBFA-1	LFA-2	LCA-2	LPCA-2	LBFA-2
+2	24.3				14.4			
+1	6.4				7.6			
+0.63	4.4				4.4			
+0.40	3.5	16.4			5.3	1.6		
+0.16	14.0	13.8			27.8	11.4		
+0.10	14.8	16.4			15.1	15.0		
+0.071	14.7	18.4	0.5	0.5	10.1	18.0	0.4	0.8
+0.045	14.8	26.8	3.5	1.0	10.0	33.0	8.4	6.0
- 0.045	3.1	8.2	96.0	98.0	5.3	21.0	91.2	93.2

Table 4. Content of Free CaO, %

Fractional class, mm	Sample							
	LFA-1	LCA-1	LPCA-1	LBFA-1	LFA-2	LCA-2	LPCA-2	LBFA-2
+2	31.6				26.3			
+1	27.0				17.8			
+0.63	28.0				19.6			
+0.40	32.9	27.3			17.2	14.4		
+0.16	23.4	20.2			12.1	11.4		
+0.10	18.6	16.8			17.5	16.0		
+0.071	10.5	11.0			14.1	13.4		
+0.045	8.9	9.1	7.9	11.2	10.7	9.6	4.3	11.2
- 0.045	10.3	11.5	5.6	10.9	8.2	7.7	3.1	8.1
Median	23.8	19.0	6.6	11.8	20.0	14.2	3.1	8.0

Table 5. Content of Mineral CO₂, %

Fractional class, mm								
	LFA-1	LCA-1	LPCA-1	LBFA-1	LFA-2	LCA-2	LPCA-2	LBFA-2
+2	4.82				0.57			
+1	2.32				0.56			
+0.63	1.60				0.54			
+0.40	1.21	1.61			0.29			
+0.16	0.68	0.78			0.23	0.36		
+0.10	0.63	0.48			0.48	0.33		
+0.071	0.50	0.34	4.64		0.22	0.24	5.00	5.22
+0.045	0.46	0.34	4.54	2.20	0.19	0.20	4.52	2.69
- 0.045	0.77	0.46	4.71	3.85	0.21	0.18	5.63	2.35
Median	3.35	1.70	4.66	3.76	0.36	0.22	5.38	2.45

Table 6. Specific Surface Area, m²/g

Fractional class, mm	Sample							
	LFA-1	LCA-1	LPCA-1	LBFA-1	LFA-2	LCA-2	LPCA-2	LBFA-2
+0.40	3.69	5.02				3.52		
+0.16	2.52	4.13				2.52		
+0.10	1.77	3.70			1.42	4.54		
+0.071	1.57	1.93			1.42	2.01		
+0.045	1.24	1.84	2.74	6.19	1.08	1.84	2.63	2.13
-0.045	2.61	2.42	3.81	5.23	1.13	1.64	1.78	2.79
Median	1.91	3.12	4.11	6.65	1.09	2.84	2.42	2.64

Table 7. Content of Sulfur, %

Sample	S _{total}			S _{sulfate}			S _{sulfide}		
	GC*1	IC*2	HAL*3	GC*1	IC*2	HAL*3	GC*4	IC*5	HAL*5
LFA-1	6.35	5.96	4.50	6.31	6.03	4.50	0.04	-0.07	0
LCA-1	7.18	6.75	6.96	7.18	6.88	6.96	0.04	-0.13	0
LPCA-1	2.71	2.66	2.67	2.53	2.70	2.67	0.05	-0.04	0
LBFA-1	2.01	1.98	2.06	1.92	2.00	2.06	0.05	-0.02	0
LFA-2	3.97	3.91	4.03	3.94	3.94	3.87	0.04	-0.03	0.16
LCA-2	5.27	5.06	5.27	5.22	5.10	5.27	0.04	-0.04	0
LPCA-2	3.02	2.96	2.96	2.75	3.03	2.96	0.05	-0.07	0
LBFA-2	2.18	2.06	2.23	2.12	2.14	2.22	0.05	-0.08	0.01

*1 Geological Centre of Estonia.

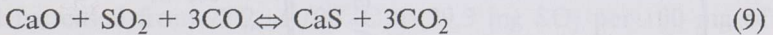
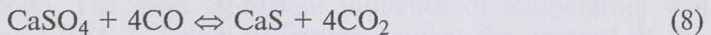
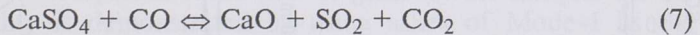
*2 Institute of Chemistry of Tallinn Technical University.

*3 Hans Ahlström Laboratory.

*4 Analysed.

*5 Calculated.

At higher temperatures (PF conditions) the binding process follows the reactions (1), and (3)-(6) and sulfur exists in the ashes only in sulfate form [12]. At lower temperatures (CFBC conditions), the formation of CaS by reaction (2) is also possible. The formation of CaS in the reducing zones of the combustion chamber would take place, according to [13-15] as follows:



The results of the chemical analysis of the samples confirmed the absence of soluble sulfide sulfur (CaS) in fly ashes (LCA, LPCA, LBFA). Not less important is the practical absence of S²⁻ ion in LFA, as the residence time of it in the oxidising conditions was shorter. The results that we obtained correlated with those obtained in the Hans Ahlström Laboratory [16] and were confirmed by the results of the control analyses

carried out at the Institute of Chemistry of Tallinn Technical University, and at the Geological Centre of Estonia (Table 7). The content of sulfide sulfur calculated as: $S_{\text{sulfide}} = S_{\text{total}} - S_{\text{sulfate}}$ was in the range of analysis error and, first of all, the value of 0.04-0.05 % could describe the possible remaining content of pyritic sulfur. The control determination of sulfide sulfur by absorbing H_2S with the following precipitation of sulfide ion as CdS [17] confirmed the absence of CaS in the ashes studied. Evidently, the experimental conditions did not favour the formation of CaS by reactions (8) and (9) but did favour the formation of CaSO_4 by reaction (6).

Reactivity of Ashes with SO_2

The influence of the feeding mode of the boiler on the SO_2 binding ability of ashes was studied at 800°C .

Ash samples bound SO_2 actively, and the binding capacity of SO_2 was high. Thus, at two- and ten-minute contact they bound 17-34 and 21-37 mg SO_2 per 100 mg of sample, respectively, and the SO_2 binding rate during the first seconds of contact was 0.38-0.47 mg SO_2/mg of sample per min (Fig. 2).

The SO_2 binding ability of furnace and cyclone ash was higher at Mode-2, of precipitation chamber and bag filter ash at Mode-1. It was caused by a comparatively finer fractional composition of samples LFA-2 and LCA-2 and they were less saturated with sulfur than similar samples

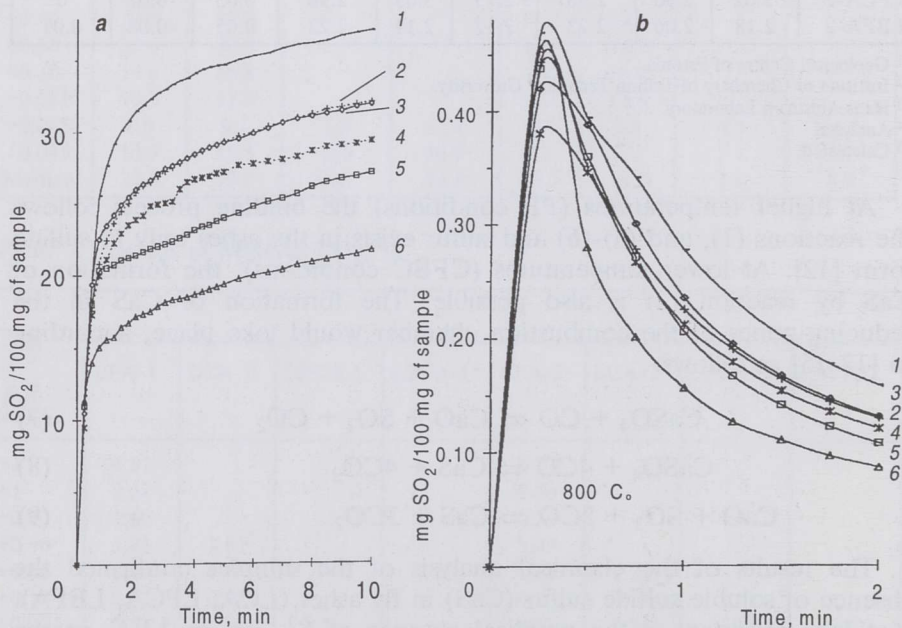


Fig. 2. SO_2 binding capacity (a) and binding rate (b) of low-temperature ashes at 800°C : 1 - LBFA-1; 2 - LBFA-2; 3 - LBCA-1; 4 - LFA-2; 5 - LFA-1; 6 - LCA-1

of Mode-1. The content of fraction $-45 \mu\text{m}$ in LFA-1 and LCA-1 was 3.1 % and 8.0 %, compared to 5.3 % and 21.0 % for LFA-2 and LCA-2 (Tables 2 and 3), respectively; the content of SO_4^{2-} in LFA-1 and LCA-1 was 18.4 % and 23.2 % towards 13.4 % and 18.0 % for these samples of Mode-2 (Table 2).

LPCA-1 and LBFA-1 bound at 10-minute contact 31.7 and 36.9 mg SO_2 per 100 mg of sample and LPCA-2 and LBFA-2 30.1 and 31.9 mg SO_2 per 100 mg of sample (Fig. 2a). The SO_2 binding rate was for LPCA-1 and LBFA-1 0.42 and 0.47 mg SO_2/mg of sample per min and for LPCA-2 and LBFA-2 0.53 and 0.44 mg SO_2/mg of sample per min, respectively (Fig. 2b).

Comparing the samples of LPCA and LBFA, there was no great difference in the content of SO_4^{2-} , but the content of the fraction $-45\mu\text{m}$ and the content of free CaO were higher, and the BET surface area was larger for samples of Mode-1 (Table 2). By using samples of LPCA and LBFA with the lowest content of free CaO, the silicates also took intensively part in the SO_2 binding process. The role of Ca-silicates and other alkali compounds of ashes as SO_2 binders is well demonstrated in Fig. 3. For the initial samples of LFA-1 and LCA-1 at 800°C and 10-minute contact, 55-60 % of the amount of free CaO was used up, considering its maximum SO_2 binding capacity. For LFA-1G this value was 117 %, and for LFBA-1 almost 137 % (or 40 and 70 %, respectively, considering the content of total CaO in these samples of ashes).

The influence of temperature and that of grinding the samples on the SO_2 binding ability is demonstrated by using ashes of Mode-1 as the main feeding mode. The most significant influence of temperature was observed by using LBFA-1. Upon 10-minute contact the amount of the SO_2 bound increased from 27.2 at 700°C to 39.3 mg SO_2 per 100 mg of samples at 950°C (Fig. 4a), or the increase was 44.5 %. It means that Ca-silicates (Table 2) bind SO_2 preferably at higher temperatures. By using the sample of LFA-1 the increase in the temperature from 700 to 950°C increased the amount of the SO_2 bound by 21 %. Grinding increased the SO_2 binding capacity of LFA-1 at 700°C and at 10-minute contact from 24.0 to 43.8 mg SO_2 per 100 mg of sample, or the increase was 55 % (Fig. 3a). For samples of LTA without previous grinding

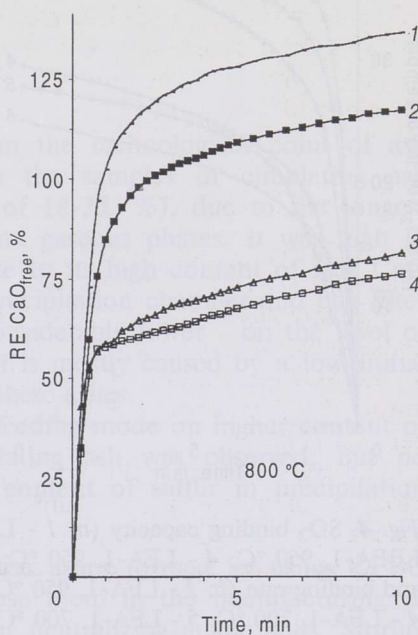


Fig. 3. SO_2 removal efficiency (RE) of free CaO of different ashes: 1 - LBFA-1; 2 - LFA-1G; 3 - LCA-1; 4 - LFA-1

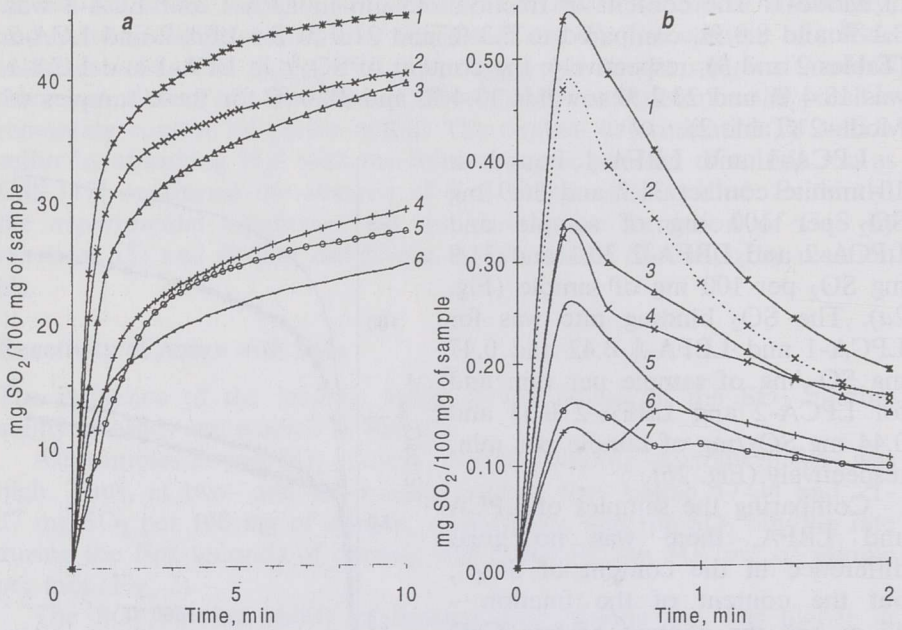


Fig. 4. SO₂ binding capacity (a: 1 - LFA-1G, 950 °C; 2 - LFA-1G, 700 °C; 3 - LBFA-1, 950 °C; 4 - LFA-1, 950 °C; 5 - LBFA-1, 700 °C; 6 - LFA-1, 700 °C) and binding rate (b: 1 - LFA-1, 950 °C; 2 - LFA-1, 800 °C; 3 - LBFA-1, 950 °C; 4 - LFA-1, 700 °C; 5 - LFA-1, 700 °C; 6 - LFA-1, 950 °C; 7 - LBFA-1, 700 °C) of ashes at different temperatures

(activation) as well as for samples of HTA [11] at higher temperatures (>900 °C) during the first seconds of interaction between gaseous and solid phases the decrease in the activity of SO₂ binding was observed. For example, the maximum SO₂ binding rate for LFA-1 at 950 °C was 0.16 mg SO₂/mg of sample per min, compared to 0.25 at 700 °C (Fig. 4b), at 800 °C it was 0.44 mg SO₂/mg of sample per min. Obviously, it could be

explained by the decrease in the specific surface area as a result of partial sintering of ashes at these temperatures.

Comparing the SO₂ binding capacity of different samples of LTA at 700 °C with that obtained with the samples of high-temperature CA and natural limestone, it was detected that the initial samples of LTA bound twice as much SO₂

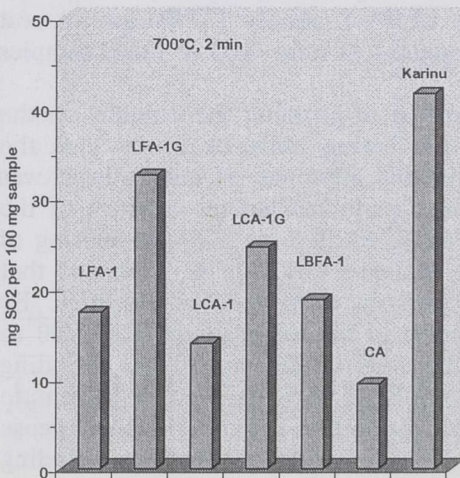


Fig. 5. SO₂ binding capacity of lime-containing materials at 700 °C after 2 min contact

(17-19 mg SO₂ per 100 mg of sample) as the initial samples of HTA (9 mg SO₂ per 100 mg of sample) or 50 % of the amount bound by the limestone of Karinu deposit. The previous grinding of samples of LFA and LCA increased their SO₂ binding capacity to the level of 80 %, compared with that of natural limestone (Fig. 5).

Conclusions

1. The content of sulfur depends on the technological point of ash sampling. It was the highest in the samples of circulating ash (expressed as SO₄²⁻ on the level of 18-23 %), due to the longest contact time between the solid and gaseous phases. It was high in furnace ash as well (13-18 %), due to its high content of free CaO and the long residence time. In precipitation chamber and bag filter ashes the content of SO₄²⁻ was considerably lower - on the level of 10 % and 7 %, respectively, which is mostly caused by a low initial content of calcium compounds in these ashes.

A positive influence of the full feeding mode on higher content of sulfur in furnace ash and circulating ash was observed, but no differences were detected in the content of sulfur in precipitation chamber and bag filter ashes.

2. The absence of sulfide sulfur in the ashes formed by using CFBC technology makes it possible to use them in the manufacturing of concrete and in the agriculture for neutralizing acidic soils. Storing CFBC ashes in dumps is harmless for surface and ground water.
3. The SO₂ binding capacity and the binding rate for different ashes formed during combustion of Estonian oil shale in circulated fluidized bed combustor were determined. All kinds of ashes removed from the furnace had a high remaining reactivity towards SO₂.

The extensive high SO₂ binding activity of these ashes is caused by the continuously renewing surface of the ash particles due to the friction of the particles in CFB conditions, which makes the inner layers of free CaO available to SO₂. It was proved by the fact that the similar effect accompanied the grinding of LFA and LCA, which was followed by a significant increase (1.5 times) in the SO₂ binding capacity of the samples.

Silicates and other alkali compounds, in addition to the free CaO, also could take part in the SO₂ binding process by using CFB technique. So, LPCA and LBFA bound up to 37 % more SO₂ as it was calculated basing on the content of free CaO in the samples.

4. A significant increase in the SO₂ binding capacity of samples was observed by increasing the temperature from 700 up to 800 °C (30-40 % during the first minutes of contact). The raise in the temperature from 800 up to 950 °C was followed by an inessential increase in the SO₂ binding capacity. Consequently, the most suitable temperature for SO₂ binding means to be 800-850 °C.

5. The LTAs bound approximately twice as much SO_2 as the high-temperature cyclone ash. It confirmed the advantage of CFB technology, compared to PF technology in binding sulfur compounds. Considering the high SO_2 binding ability of LTA and the absence of sulfide sulfur in it, the CFBC technique might prove to be one of the most environmentally harmless techniques applicable in the future energy production in Estonia.

REFERENCES

1. *Veiderma M.* Natural resources in Estonia and the problems of utilisation // Probleme und Strategien der Umstrukturierung von Industrieregionen - Mit Blick auf Ida-Viru in Estland / P. J. Tettinger, M. Veiderma (Hrsg.). Stuttgart etc.: Boorberg, 1997. S. 1-29 (Bochumer Forschungsberichte zum Berg- und Energierecht. Bd. 19).
2. Annual Report of SE Eesti Energia 1995. Tallinn, 1996 [in Estonian].
3. Circulating Fluidized Beds / J. R. Grace, A. A. Avidan, T. M. Knowlton (Eds.). - London etc.: Blackie Academic and Professional, 1997.
4. *Abdullally I. F., Alkon I.* Advanced pressurized circulating fluidized bed system: beyond the demonstration stage // Proc. 13th Intern. Conf. on Fluidized Bed Combustion / K. H. Heinchel (Ed.) New-York: ASME, 1995. Vol. 1. P. 625-636.
5. *Prikk A., Hiltunen M., Makkonen P.* Circulating fluidized bed boilers // Oil Shale. 1997. Vol. 14, No. 3 Special. P. 254-264.
6. *Holopainen H.* Experience of oil shale combustion in Ahlström pyroflow CFB-boiler // Oil Shale. 1991. Vol. 8, No. 3. P.194-209.
7. *Öpik I.* The Chatham CFB boiler for a wide spectrum of fuels and some problems of Estonian oil shale combustion in CFB systems // Oil Shale. 1995. Vol. 12. No. 2. P. 179-184.
8. *Arro H., Prikk A., Kasemetsa J.* Circulating fluidized bed technology - test combustion of Estonian oil shale // Oil Shale. 1997. Vol. 14, No. 3 Special. P. 215-217.
9. *Öpik I., Prikk A.* The 41 MWe LLB CFB-boiler as model for 200 MWe oil-shale blocks // Oil Shale. 1996. Vol. 13, No. 3. P. 239-245.
10. *Arro H., Prikk A., Kasemetsa J.* Recommendations for design of Estonian oil shale fired CFB boilers // Oil Shale. 1997. Vol. 14, No. 3 Special. P. 246-253.
11. *Kaljuvee T., Triikkel A., Kuusik R.* Reactivity of oil shale ashes towards sulfur dioxide. 1. Activation of high-temperature ashes // Oil Shale. 1997. Vol. 14, No. 3. P. 393-408.
12. *Dam-Johansen K., Ostergaard K.* High-temperature reaction between sulfur dioxide and limestone. 4. A discussion of chemical reaction mechanisms and kinetics // Chem. Eng. Sci. 1991. Vol. 46, No. 3. P. 855-859.
13. *Lyngfelt A., Leckner B.* SO_2 capture in fluidized-bed boilers: re-emission of SO_2 due to reduction of CaSO_4 // Chem. Eng. Sci. 1989. Vol. 44, No. 2. P. 207-213.

14. Hansen P. F., Dam-Johansen K., Ostergaard K. High-temperature reaction between sulfur dioxide and limestone. 5. The effect of periodically changing oxidizing and reducing conditions // Chem. Eng. Sci. 1993. Vol. 48, No. 7. P. 1325-1341.
15. Kuusik R., Veiderma M. Thermal processing of phosphogypsum // Proc. 3rd Intern. Symp. on Phosphogypsum. Orlando, Florida, 1990. P.267-279.
16. Arro H. Research for designing fluidized bed boiler and ash deposits formed at the oil shale test burning. 1 // Report of Tallinn Technical University. Tallinn, 1995. P. 89 [in Estonian].
17. Jeffery P.G. Chemical Methods of Rock Analysis. Pergamon Press, Oxford etc., 1970.

Presented by J. Kann

Received June 27, 1998