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Combustion of Shale Heavy Coal-Tar Products in a Boiling Layer of a Catalyst

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Abstract

The work studied the catalytic combustion of heavy coal-tar products (HCTP) formed upon processing of Baltic shales to assess the efficiency of combustion in the presence of an Al–Cu–Cr oxide catalyst of complete oxidation of organic compounds with active component contents of 10 %, the optimum temperature modes of catalytic combustion were determined and an assessment of an opportunity for carrying out fuse combustion process in a boiling layer of a catalyst without using additional fuels was performed. It was demonstrated that the maximum degree of combustion of 97.7–97.8 % was reached at 700–750 °C. Herewith, the concentrations of harmful substances in exhaust gases were the following (ppm): CO 244–269; NO_x 179–229; SO_2 is absent. Based on the literature data on catalytic combustion of oil containing sulphur, it was demonstrated that the SO_2 concentration at the outlet of the reactor could reach the value of 60 ppm during long-term operations of the setup for catalytic combustion of HCTP in autothermal mode considering the calcium oxide content in their mineral component. The carbon content in bottom ash amounted to 2.3–2.4 %. By the calcium oxide content, ash refers to basic fly ash and can be used as an additive to cement. Based on the results of catalytic combustion of HCTP in autothermal mode in a laboratory setup, emission of harmful substances in exhaust gases was determined using HCTP as fuels for a catalytic boiler-house with the air excess coefficient $\alpha = 1.2$. Emissions of hazardous substances during combustion of HCTP do not exceed the value of maximum permissible emissions of each component right up to a background value of 0.9MPC.

Key words: shale fuse, catalyst, boiling layer, combustion

INTRODUCTION

The preparation of resin as the major product of processing of oil shales is one of the directions of their use in energetics [1]. Heavy coal-tar products (HCTP) are formed as byproducts during gas-generating processing of oil shale resulting from enveloping the fly solid phase with resin contained in a gas-vapour mixture [2].

To recycle HCTP such methods, as solid fuel combustion, a return to gas generators for additional processing, resin extraction from coal-tar products [2, 3], as well as joint recycling with other solid wastes by thermolysis are currently used [4]. Nevertheless, shale processing wastes are often stored, causing damage to the environment [5]. Heavy coal-tar products formed during shale processing can be recycled using catalytic combustion technologies.

Technology of fuel combustion in a boiling layer of a catalyst based on four principles was developed at the Boreskov Institute of Catalysis SB RAS (Novosibirsk): catalytic deep oxidation of organic substances, the use of the boiling bed of catalyst particles, combustion of mixtures of fuel and air close to stoichiometry, the combination of heat dissipation and heat sink in a single catalyst layer [6]. Works [7–10] demonstrate that using such technologies enables to efficiently combust various fuels, including low-calorie (peat, sawdust, oil shales, sewage sludge), herewith, the degree of fuel burn-up is over 97 %.

This work studies catalytic combustion of HCTP formed upon processing of Baltic shales with the aim of assessing combustion efficiency of an industrial catalyst for complete oxidation of organic compounds (ShchKZ-1), determining the optimum temperature modes of catalytic combustion and evaluating an opportunity to carry out the combustion process in a boiling layer of a catalyst without using additional fuels when carrying out the process in industrial scales.

EXPERIMENTAL

To study the process of catalytic combustion of HCTP in a boiling layer of an Al-Cu-Cr oxide catalyst (ShchKZ-1) manufactured by the Shchelkovo Catalyst Plant Ltd. (average particle size of 1.75 mm, active component contents of 10 mass %, TU 2171-005-51444844–01) was used. The catalyst characteristics were studied in detail in [11].

The gas composition at the outlet of the reactor was determined using the gas-analyzer Polar manufactured by the Promekopribor Ltd. (St. Petersburg, Russia).

The moisture content of HCTP was determined by the mass loss during drying at 105–110 °C until a constant mass is reached [12]. Considering that HCTP may contain organic substances that may evaporate at this temperature, moisture content was additionally determined according to the mass loss upon ageing of HCTP in a desiccator with chemically pure KOH (GOST 24363–80) for 100 h at room temperature.

The amount of volatile substances in HCTP was determined by the mass loss at 900 °C for 7 min [13].

Ash content in HCTP before combustion and the residue after combustion in the boiling layer of a catalyst was determined by the mass loss upon calcination at 815 °C in the air current for 2 h [14].

Elemental analysis of the organic component of HCTP in the ash residue after their combustion in the boiling layer of a catalyst was carried out using a Vario EL cube Elementar HCNS analyzer (Germany).

The content of elements entering into ash composition of HCTP was determined by X-ray fluorescence spectral method using an ARL-Advant'x analyzer (Switzerland) with a rhodium anode of an X-ray tube.

The degree of burn-up of HCTP (β) was calculated by the formula

$\beta = 10^4(A_s - A_0)/A_s(1 - A_0)$

where A_0 is the ash proportion in the original dry sample, %; A_s is the ash proportion in the solid product after combustion of HCTP, %.

The heat of combustion of HCTP in a bomb was determined using the IKA C200 calorimeter (Germany).

The release of gaseous harmful substances upon combustion of HCTP F1 (B_j, g/s) for a catalytic boiler-house of the power of 1 MW with excess air coefficient $\alpha = 1.2$ was determined by the formula

$B_j = B_{j/\mathrm{sp}} G_{\mathrm{HCTP}}$

where $B_j/_{\mathrm{sp}}$ is the amount of gaseous harmful substance formed upon the combustion of 1 kg of HCTP F1, g/kg; $G_{\mathrm{HCTP}} = 0.060$ kg/s is the fuel consumption for the boiler of 1 MW.

The data obtained upon the combustion of HCTP F1 in autothermal mode in a laboratory setup at 700 °C were used for determining the $B_j/_{\mathrm{sp}}$ value by the formula

$B_{j/\mathrm{sp}} = 10^{-3} C_j k_j V / g_{\mathrm{HCTP}}$

where C_j is the concentration of j-th gaseous harmful substance in exhaust gases, ppm; k_j is conversion coefficients of ppm into mg/m^3 given in [15], $V = 0.0028$ is the volume of exhaust gases during autothermal combustion of HCTP F1 in a laboratory setup at 700 °C, m^3/s; $g_{\mathrm{HCTP}} = 0.183 \cdot 10^{-3}$ is the consumption of HCTP F1 upon autothermal combustion in an autothermal mode at 700 °C, kg/s. During combustion of HCTP F1 in autothermal mode, a value of 60 ppm was used for the concentration of exhaust gases in case of SO_2.

Release of dust (R_d, g/s) was calculated in case of the sequential use for gases purification of cyclone of the SKTsN-34 type with a purification degree of 89 % [16] and a bag filter setup of the RFU3.5 type with a purification degree of no lower than 99.9 % [17] considering ash content in HCTP and the degree of their burnout at 700 °C the formula

$$R_d = 1000G_{HCTP}[A^r + (1 - A^r - W_t^r)(1 - \beta)](1 - \eta_c) \times \times (1 - \eta_f)$$

where G_{HCTP} equal to 0.060 kg/s represents fuel consumption for 1 MW boiler; $A^r = 0.398$ is ash content of working mass of HCTP in fractions; $W_t^r = 0.077$ is total moisture in HCTP in fractions; $\beta = 0.977$ is the degree of burnout of HCTP in fractions; $\eta_c = 0.89$ is the degree of purification of the gas in the cyclone into fractions; $\eta_f = 0.999$, is the degree of gas purification using a filter in fractions.

Maximum allowable emission of *j*-th gaseous harmful substance (MAE$_j$) was determined in accordance with normative documents [18]. Its value for a single sourcewith a round mouth in case of c_{HCTP} less than MPC for each component was determined by the formula

$$MAE_j = (MPC_j - c_{b/j})H^2(V_1\Delta T)^{1/3}/AFmn\eta$$

where MAE is maximum allowable emission of harmful substances, g/s; MPC is the maximum permissible concentration of harmful substances, mg/m^3 (single MPCs are used in the calculation); $c_{b/j}$ is a various background concentrations of harmful substances; $H = 20$ m is the height of the emission source above ground level; $V_1 = 0.353$ is the volume of flue gas, m^3/s; $\Delta T = 94$ °C is the temperature difference between the temperature of emitted gas mixture $T_g = 120$ °C and the temperature of the ambient air $T_a = 26$ °C (when determining the value of ΔT the temperature of the ambient air T_a should be taken equal to the average maximum outside temperature of the most hot month of the year); A is a coefficient depending on temperature stratification of the atmosphere (the values for different regions are given in [18], for the European territory of Russia to the north of the 52° north latitude ($A = 160$); F is a dimensionless coefficient that takes into account the rate of sedimentation of harmful substances in ambient air (gaseous harmful substances and fine aerosols, such as dust and ash, the rate of orderly sedimentation of which is almost equal to zero, $F = 1$); $n = 1.82$, $m = 0.43$ are dimensionless coefficients that take into account the terms of exit gas mixture from the mouth of the emission source (calculated by the formulas given in [18]; η is the dimensionless coefficient that takes into account the effect of the terrain (in case of a flat or moderate ter-

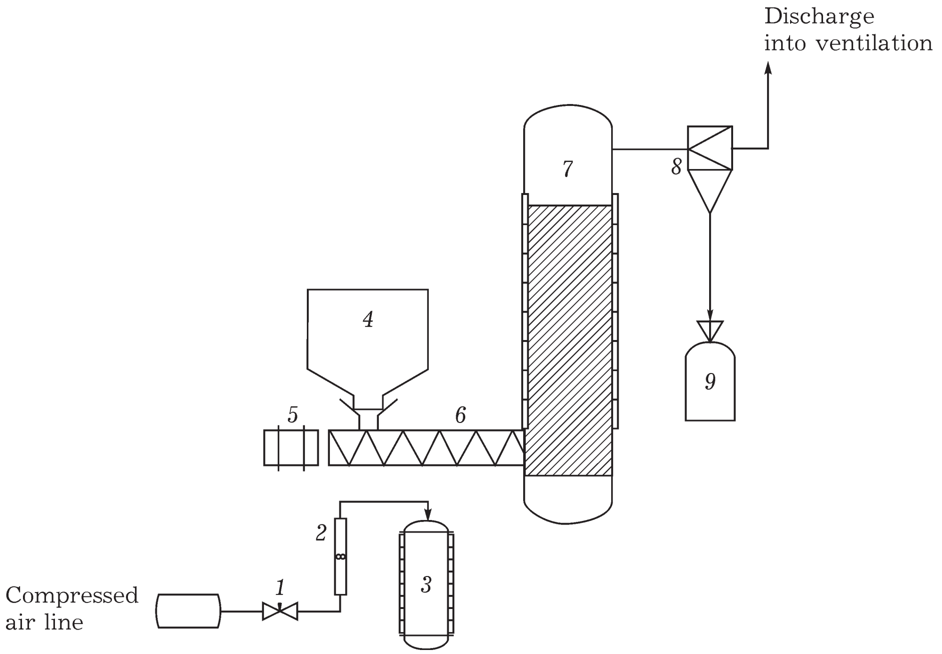

Fig. 1. Schematic diagram of the installation to study the process of catalytic combustion of HCTP: *1* – control valve, *2* – rotameter, **3** – electric heater, *4* – bunker for solid fuels (HCTP), *5* – electromotor, *6* – auger meter, *7* – fluidized bed reactor with electric heating, *8* – cyclone, and *9* – container for ash.

rain with a height difference of no more than 50 m per 1 km is equal to 1); the MPC values of harmful substances in the calculations are adopted in accordance with hygienic standards [19]: maximum single $\text{MPC}_{SO_2} = 0.5\ \text{mg/m}^3$, MPC_{NO_2} = of $0.085\ \text{mg/m}^3$, $\text{MPC}_{NO} = 0.4\ \text{mg/m}^3$, $\text{MPC}_{CO} = 5\ \text{mg/m}^3$, $\text{MPC}_{dust} = 0.3\ \text{mg/m}^3$ (for dust with silica content of 20–70 %).

A scheme of a setup for the study of catalytic combustion of HCTP is presented in Fig. 1.

Into reactor (*7*) with an internal diameter of 75 mm and a height of 1500 mm made of stainless steel, 3.5 L of a catalyst for complete oxidation of organic compounds ShchKZ-1 was loaded. HCTP (F1) preliminarily crushed to the particle size of less than 5 mm were loaded into bunker (*4*). To output the reactor in operating temperature mode, a layer of a catalyst in boiling mode was heated with an external electric heater of the reactor and an air electric heater (*3*) at an air flow 10 m^3/h. The air flow was set by means of valve (*1*) and rotameter (*2*). The resulting residue from combustion of HCTP in the mode of pneumatic transport went into cyclone (*8*) and after separation from the gas, entered into a container for ash (9). The combustion process was carried out at temperatures of 650, 700 and 750 °C. Consumption of HCTP (F1) was 610–710 g/h.

RESULTS AND DISCUSSION

Samples of Baltic HCTP F1 and F2 used in the work were of various consistencies.

HCTP were in the form of lumps that were crumbled into smaller particles. The density of HTCP F1 in such a form was 1050 kg/m^3. HCTP F2 species were in the form of a single piece with a density of 1250 kg/m^3. They cracked upon mechanical effects but were not separated into separate particles, showed plasticity. Such a difference in properties is apparently related to different contents of the solid phase and water. Considering different properties of F1 and F2 their elemental composition, ash content and moisture were determined. Mass loss in F1 upon drying at 105–110 °C was 14.5 %, in F2 – 17.8 %. A high value of moisture is apparently related to the release of volatile organic compounds (VOCs) at this temperature. Determining the moisture content using drying in a desiccator at room temperature to exclude the release of VOCs demonstrated that mass loss was

TABLE 1

Working mass composition of heavy coal-tar products (HTCP) F1 and F2

HTCP	Working mass composition, %					A^r	W_t^r, %***
samples	C^r	H^r*	O^r**	S^r	N^r		
F1	40.0	4.1	7.0	1.3	0.1	39.8	7.7
F2	40.5	3.9	8.3	1.2	0.1	34.8	11.2

*The hydrogen content was determined by the formula: $H^r = H^a - 0.112W^r_t$, where H^a is the hydrogen content in the analysis sample, %; W_t^r is total moisture in heavy HTCP, %; 0.112 is a coefficient taking into account the hydrogen content in water; index "r" indicates the operating status of the fuel.

**The oxygen content was determined from the balance.

***The amount of total moisture is taken by the mass losses during drying in a desiccator over KOH.

TABLE 2

Composition of dry benzene mass of heavy coal-tar products (HTCP) F1 and F2

HTCP	V^{daf}, %	Composition of dry benzene mass, %				
samples		C^{daf}	H^{daf}	O^{daf}	S^{daf}	N^{daf}
F1	84.2	76.2	7.8	13.3	2.5	0.2
F2	86.5	75.0	7.2	15.4	2.2	0.2

TABLE 3

Heat of combustion of coal-tar products (HCTP)

Parameters	Value	
	kJ/kg	kcal/kg
Heat of combustion of F1 in a bomb (Q_b^a)	18 243	4357
Highest Heat of Combustion of F1 (Q_s^a)	18 093	4321
Lowest heat of combustion of F1 (Q_i^a)	17 010	4063
Lowest heat of combustion of F1 (Q_i^r) by Mendeleev's formula[23]	16 974	4054
Lowest heat of combustion of F2 (Q_i^r) by Mendeleev's formula [23]	16 677	3988

7.7 % in F1, and 11.2 % in F2. Thus, compounds volatile at 110 °C in the amount of 6.8 % in F1 and 6.6 % are a part of the composition of HTCP.

Working mass composition of F1 and F2 is presented in Table 1, the combustible mass – in Table 2. The composition of dry benzene mass of HTCP by formulas given in [20]. Their heats of combustion are provided in Table 3.

The highest heat of combustion Q_s^a was determined according to [21, 22] by the formula

$$Q_s^a = Q_b^a - (94\,S_t^a + \alpha Q_b^a)$$

where 94 is a coefficient taking into account the heat of formation of sulphuric acid from sulphur dioxide and dissolution of sulphuric acid in water per 1 % of sulphur, passed from fuel combustion into sulphuric acid, kJ/kg; S_t^a is the mass fraction of sulphur in the fuel; α is the coefficient taking into account the heat of formation and dissolution in water of nitric acid equal to 0.0015 for shales.

The lowest heat of combustion Q_i^a was calculated by the formula

$$Q_i^a = Q_s^a - 24.42(8.94H^a + W^a)$$

where 24.42 is the heat of evaporation at a temperature of measurements of 25 °C at a rate of 1 % of isolated water, kJ/kg; 8.94 is the conversion rate of the mass fraction of hydrogen for water; H^a is the mass fraction of hydrogen in analysis sample of fuel; W^a is the mass fraction of water in analysis sample of fuel.

The lowest heat of combustion Q_i^r (kcal/g) was also calculated by Mendeleev's formula given in work [23]:

$$Q_i^r = 81C^r + 246H^r - 26(O^r - S^r) - 6\,W_t^r$$

From the results obtained it follows that the lowest heat of combustion of analysis sample of HCTP F1 Q_i^a is in good agreement with the lowest heat of combustion of HCTP F1 Q_i^r calculated by Mendeleev's formula.

Ash (incombustible residue) content obtained at 815 °C and air blowing of a muffle furnace is given in Table 4.

Absorber additives, for example, those of calcite are used on an industrial scale in a boiling layer upon combustion of fuels containing sulphur. To efficiently capture SO_2 the molar ratio of Ca/S should be no less than 2 [24]. Assessment of this ratio in HCTP demonstrates that Ca/S = 2.6 for F1, Ca/S = 2.9 for F2. Thus, it can be suggested that one does not need introduce an additional sulphur absorber. By calcium oxide content, ash refers to basic fly ash

TABLE 4

Ash contents of heavy coal-tar products (HCTP), %

Components	HCTP F1	HCTP F2
Na_2O	0.91	0.53
MgO	5.31	5.48
Al_2O_3	17.44	16.89
SiO_2	45.72	45.86
P_2O_5	0.37	0.31
SO_3	4.17	3.41
Cl	0.07	0.18
K_2O	4.71	3.41
CaO	14.78	17.51
TiO_2	0.56	0.54
Fe_2O_3	5.49	4.27
ZnO	0.16	0.05
SrO	0.07	–
BaO	0.06	–
Other compounds	0.18	1.56
Total balance	100	100

Note. Dash indicates is absent.

TABLE 5

Results of combustion of coal-tar products (HCTP) F1 in a boiling layer of ShchKZ-1 catalyst in autothermal mode at various temperatures

T, °C	F1 consumption, kg/h	Air consumption, nm^3/h	Residual ash content, %	Carbon content in ash, %	Degree of burnout of F1, %
650	0.61	10.0	95.80	3.00	96.7
700	0.66	10.0	97.09	2.30	97.7
750	0.71	10.0	97.16	2.40	97.8

and can be used as an additive to cements [25], as well as fly ash formed during combustion of various coals [26].

The experimental setup on combustion of HCTP was equipped with a screw feeder that is designed to dose bulk fuels. In this regard, HTCP F1 was selected for work. After grinding of pieces up to the sizes of less than 5 mm, a fairly loose mixture of particles was formed. The apparent density of crushed HCTP F1 was 788 kg/m^3.

Table 5 gives the results of combustion of HCTP F1 in a boiling layer of an Al–Cu–Cr oxide catalyst in autothermal mode at various temperatures. The content of hazardous substances at the outlet of the reactor is given in Table 6. From the results presented in Tables 5 and 6, it follows that the maximum degree of burnout of 97.7–97.8 % is reached at process temperature of 700–750 °C, herewith, the concentrations of hazardous substances in exhaust gases were as follows (ppm): CO 244–269, NO_x 179–229, SO_2 is absent. Carbon content in ash residue was 2.3–2.4 %.

Work [27] demonstrated on an example of catalytic combustion of sulphur and high-sulphur crude oil in a boiling layer of the same Al–Cu–Cr oxide catalyst that sulphur accumulation in a catalyst to a certain amount occurred in the initial time and the SO_2 concentration in exhaust gases was equal to zero. The concentration at the outlet of the reactor in this case went out to a constant value after three hours of work. Upon addition of calcium calcite or calcium oxide (with a Ca/S molar ratio of 2.0), the SO_2 concentration was 10 % of the maximum possible value that corresponded to the transition of entire sulphur contained in combustible mass into SO_2. In this regard, it should be expected that an increase in the SO_2 concentration in exhaust gases will occur during long catalyst operation upon combustion of HCTP after saturation of the catalyst with sulphur. Considering attenuation range of SO_2 by CaO contained in inorganic component of HTCP equal to 90 % at 700 °C in the combustion zone, the SO_2 concentration in exhaust gases upon combustion of HTCP F1 in an autothermal mode can reach a value of 60 ppm. Calcium sulphate formed in the end during the interaction of calcium oxide with SO_2 in the presence of oxygen under these conditions is almost not decomposed, because decomposition of calcium sulphate begins at temperatures above 1000 °C [28].

From the results obtained it follows that technology of catalytic combustion of fuels in a boiling layer can be used to dispose HTCP in autothermal mode with achieving a high de-

TABLE 6

Content of hazardous substances at the outlet of the reactor (air consumption of 10.0 nm^3/h)

T, °C	F1 consumption, kg/h	Content, ppm				
		NO	NO_2	CO	SO_2	CH_4*
650	0.61	153	0	230	0	0
700	0.66	178	1	244	0	0
750	0.71	225	4	269	0	0

*In vol. %.

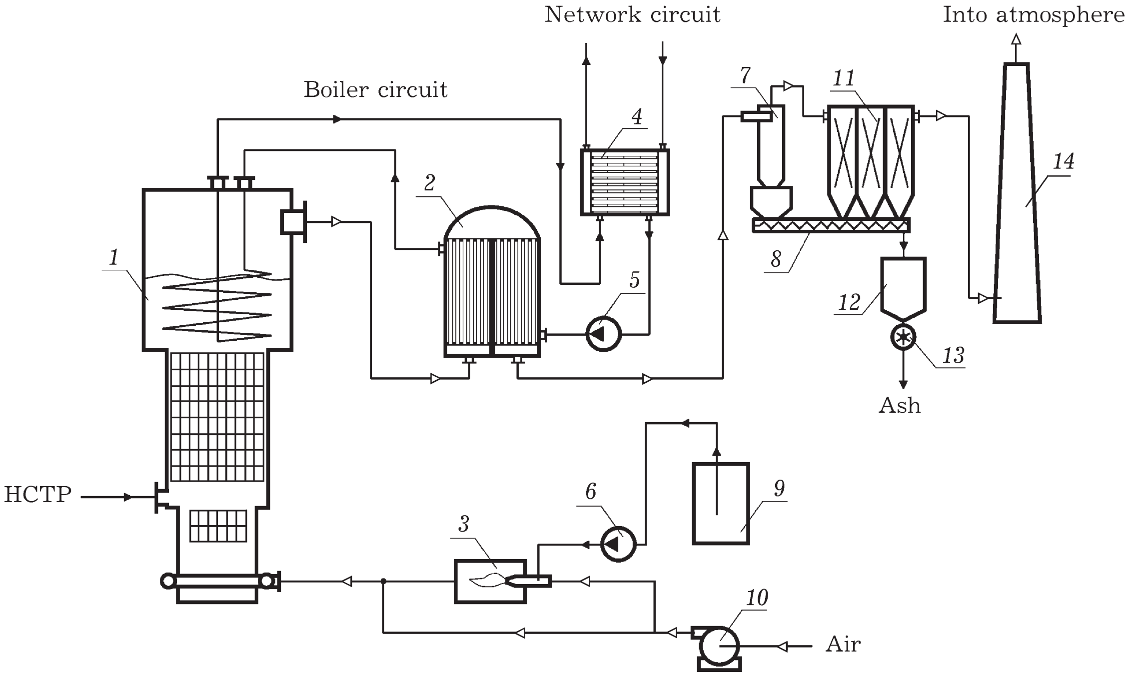

Fig. 2. Principal scheme of catalytic combustion of HCTP: *1* – reactor boiling catalyst layer, *2* – economizer, *3* – liquid-fuel heat generator for preheating the catalyst bed during the start-up period, *4* – heat exchanger, *5* – circulating pump of the boiler circuit, *6* – pump for feeding liquid fuel, *7* – cyclone, *8* – ash transporter, *9* – liquid fuel tank, *10* – blower unit, *11* – bag filter, *12* – ash bunker, *13* – gateway feeder, *14* – chimney.

gree of burnout. The heat released during combustion of HTCP can be used to provide an autonomous heat supply of residential and industrial buildings with thermal energy and hot water. Herewith, fuel combustion is carried out at the air excess factor $\alpha = 1.2$. Figure 2 demonstrates a flow sheet of catalytic combustion of HTCP with obtaining hot water for heat supply. The scheme description and working principles of the setup are given in [9].

When implementing combustion technology on an industrial scale, issues of environmental compatibility of technologies are relevant. In this regard, calculations of emission of hazardous substances into the atmosphere for a catalytic boiler with a power of 1 MW operating on HTCP. Table 7 gives the values of emissions of harmful substances during catalytic combustion of HTCP F1 at 700 °C calculated for a boiler of a power of 1 MW with the air excess coefficient $\alpha = 1.2$ based on the results of combustion of HTCP F1 in autothermal mode in a laboratory setup and maximum allowable emissions (MAE) at various background concentrations of

TABLE 7

Values of emission of harmful substances during catalytic combustion of coal-tar products (HCTP) F1 at 700 °C and maximum allowable emissions (MAE) with various background concentrations of hazardous substances ($c_{b/j}$)

Substances	MAE$_j$, g/s, with $c_{b/j}$			Emission during catalytic combustion of HCTP B_j, g/s
	0	0.5 MAE	0.9 MAE	
NO	4.08	2.04	0.41	0.21
NO_2	0.86	0.44	0.09	$1.88 \cdot 10^{-3}$
CO	51.0	25.5	5.10	0.28
SO_2	5.10	2.55	0.51	0.16
Dust	3.05	1.53	0.31	$2.71 \cdot 10^{-3}$

harmful substances ($c_{b/j}$) at emission height of 20 m, an exhaust gas temperature of 120 °C, the rate of gas outlet from the mouth of 20 m/s.

Thus, emission of harmful substances during catalytic combustion of HTCP in a boiling layer does not exceed MAE even with a background value of the concentration of components at a level of 0.9MPC.

CONCLUSION

Parameters of the combustion process of shale heavy coal-tar products (HCTP) representing a mixture of fly ash and resin in a boiling layer of an Al–Cu–Cr oxide catalyst with 10 % active component contents in autothermal mode were determined. It was demonstrated that the maximum degree of burnout of heavy coal-tar products (HCTP) of 97.7–97.8 % was reached at 700–750 °C. Herewith, the concentrations of hazardous substances in exhaust gases were as follows (ppm): CO 244–269, NO_x 179–229, SO_2 is absent. Based on literature data analysis of catalytic combustion of oil containing sulphur, it was demonstrated that during long-term operation of asetup for catalytic combustion of shale heavy coal-tar products (HCTP) in an autothermal mode with consideration of CaO content in the mineral component of HCTP, the SO_2 concentration at the outlet of the reactor could reach a value of 60 ppm. Carbon content in the ash residue was 2.3–2.4 %. By CaO content, ash refers to basic fly ash and can be used as an additive to cement.

Based on the results of catalytic combustion of HCTP in autothermal mode in a laboratory setup, emission of hazardous substances in exhaust gases was determined when using HCTP as fuels for a catalytic boiler with the air excess coefficient $\alpha = 1.2$. It was demonstrated that emissions of hazardous substances during combustion of HCTP did not exceed the values for maximum allowable emissions (MAE) of each component up to a background value of 0.9MPC.

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