CATALYSIS

Burning of Sulfur-Containing Liquid Fuels in Fluidized Catalyst Bed

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Abstract—Results are presented of the process of burning of sulfur-containing liquid fuels in a nonisothermal fluidized bed of a ShchKZ-1 industrial catalyst for the example of sulfurous and heavy high-sulfur oil. The optimal ("dropdown") temperature profile in the bed was determined. This profile is characterized by a downstream decrease in temperature from 700 to 500°C. A mathematical simulation based on the description of experimental data on the variation of the sulfur concentration at the exit from the adsorbent (calcite) bed at its varied charge was used to calculate the specific capacity of the sorbent to be $(14.2–15.9) \times 10^{-3}$ g cm^{-3}. It was shown that the potential capacity of calcite in fluidized bed exceeds by an order magnitude that in a fixed bed.

Keywords: catalytic burning, fluidized bed, sulfur-containing liquid fuels, sulfur oxides, calcite, simulation

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The technology of the catalytic burning of fuels in a fluidized bed [1] is based on the combination of four principles: use of full-oxidation catalysts, use of a fluidized bed of catalyst particles, burning of nearly stoichiometric mixtures of a fuel and air, and combination of heat release and heat removal in the same catalyst bed. The presence of a deep-oxidation catalyst in the reactor lowers the burning temperature of the organic fuel from 1200–1600 to 400–700°C, with higher oxidation rates and complete burning of fuel–air mixtures thereby provided. The catalytic burning differs fundamentally from the combustion in the conventional sense because the fuel present in the gas phase is oxidized on the surface of solid catalysts without forming a flame.

One of problems associated with the burning of various fuels is the formation of sulfur oxides, dangerous environment contaminants. However, this problem can be eliminated in the case of burning of coals (the ash of which contains such substances as SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, TiO_2, K_2O, Na_2O, SO_3, P_2O_5 [2]) because of the binding of sulfur oxides being formed by metal oxides.

In addition, it is known that some components of the flue ash, which contain 30–92% iron oxide in various forms, can exhibit a catalytic activity in oxidation processes [3–6]. Therefore, it would be expected that SO_2 is partly oxidized to SO_3, with the subsequent interaction with the oxides [7–9] in burning of coal in a fluidized bed. It was shown in [10] that, in this case, the ash with high content of CaO provides an about 80% degree of binding of sulfur oxides at Ca/S = 2, and raising the Ca/S ratio leads to a larger degree of binding of sulfur oxides.

Thus, the burning of high-ash coals with high content of CaO in the fluidized catalyst bed yields SO_2 and, partly, SO_3, with additional oxidation of SO_2 to SO_3 on the catalyst, followed by binding of sulfur oxides by the mineral part of the coals.

The content of ash in liquid fuels, such as oil, does not exceed tenths of a percent [11]. Therefore, it is necessary to additionally introduce alkaline additives into the reaction medium for effective catching of sulfur oxides. This can be done with, e.g., calcite, dolomite, montmorillonite, and calcium oxide [12–16], which are widely used to remove sulfur oxides, including the case of effluent gases in burning of both solid and liquid fuels.

Calcite has found the widest application for SO_2 binding, e.g., in burning of fuels in a fluidized bed of an
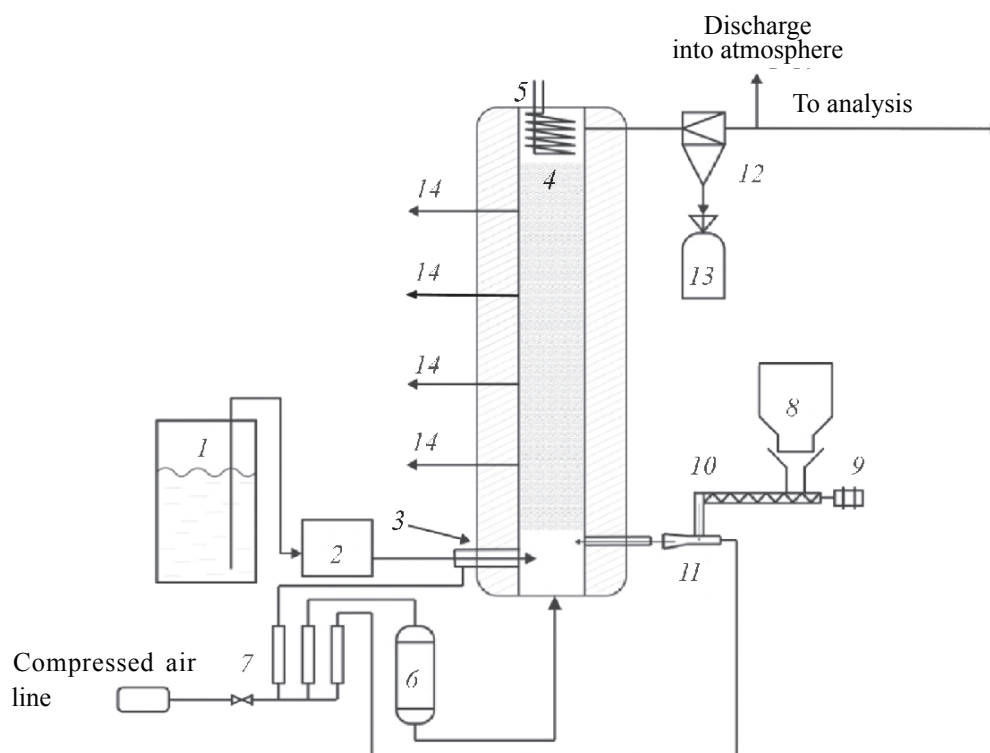
Fig. 1. Schematic of installation for burning of liquid fuels in a boiling catalyst bed. (*1*) Vessel with oil, (*2*) pump, (*3*) nozzle, (*4*) reactor, (*5*) heat exchanger, (*6*) air heater, (*7*) rotameters, (*8*) bunker for binding of sulfur compounds, (*9*) batching unit drive, (*10*) screw batching unit, (*11*) ejector, (*12*) cyclone, (*13*) vessel for collection of solid particles, (*14*) sampling devices.

inert material (sand) [12]. In oxidation of fuels, sulfur compounds are oxidized to SO_2. Sulfur oxides are absorbed in the presence of calcite.

In the case under consideration, the physical pattern of the process is, in contrast to the burning of fuels in a fluidized bed of an inert material, fundamentally different: in a reactor with a fluidized catalyst bed, there occurs catalytic burning of sulfur-containing liquid fuels to give SO_2, with its additional oxidation to SO_3 [12]. Accordingly, calcium sulfate is additionally formed via a direct interaction of calcite with SO_3.

The goal of our study was to examine the burning of sulfur-containing liquid fuels in a reactor with a nonisothermal fluidized bed of an industrial deep-oxidation catalyst in the presence of calcite and to optimize the temperature profile in the bed.

EXPERIMENTAL

To perform experiments, we used sulfurous oil (SO) and high-sulfur heavy oil (HO). The content of sulfur in the oils was determined by X-ray fluorescence analysis

[GOST (State Standard) R 51947–2002, ASTM D-4294–98}. The elemental composition of oil was determined with a VARIO EL CUBE CHNS analyzer manufactured by Elementar Analysensysteme GmbH, Germany. The main characteristics of the oils are presented in Table 1.

Oil was burnt in an installation shown schematically in Fig. 1. The installation comprised a vessel with a liquid fuel delivered by a pump into a reactor with diameter of 40 mm and a fluidized bed through an air-blown side aperture in the lower part. Also, air heated to 500°C by an air heater is delivered from below into the reactor. The air is heated for preliminary heating of the reactor and putting it into operation. As the flow rate of oil increases, the heating temperature of air entering the reactor becomes lower and the heating is fully switched off when the autothermal mode of fuel burning is reached. The air flow rate is controlled by rotameters. The reactor is also equipped by a system for delivery of the solid sulfur binder with a screw batching unit after the reactor, the ash and binder particles are separated from effluent gases in a cyclone and delivered into a vessel for collection of solid particles. Liquid fuels were burnt in a bed of ShchKZ-1 aluminum–

Table 1. Main characteristics of oils used in the study

Oil	Density, g cm^{-3} (20°C)	C	H	O	N	S
		wt %				
Sulfurous oil	0.840	84.60	12.58	0.65	1.51	0.66
High-sulfur heavy oil	0.872	84.72	12.53	0.16	0.29	2.30

copper–chromium catalyst (TU 2171-005-51444844–01, Shchelkovo Catalyst Plant OOO) having the form of spherical particles with grain size of 1.5–2.0 mm. The fraction of the active component in the catalyst was 10% (6.5 Cr_2O_3 and 3.5% CuO). The specific surface area of the catalyst was 115 m^2 g^{-1}. The catalyst was charged in an amount of 700 cm^3. The copper–chromium systems are widely used as deep-oxidation catalysts. In addition, they are rather stable against poisoning by sulfur oxides [17].

Experiments on burning of liquid fuels were performed in the modes isothermal and nonisothermal along the bed height at a burning temperature of 700°C. The nonisothermal temperature profile was created in the reactor by a heat exchanger submerged in the catalyst bed. The height of the fluidized bed was 950 mm, the fuel-burning zone in which the temperature of 700°C was maintained was 600 mm wide. The air flow rate was 2.6–2.9 m^3 h^{-1}, and the oil expenditure, 150–350 g h^{-1}.

As the binding agent for sulfur oxides served calcite (TU 8294-003-55768952–05) more than 90% composed of CaO. The bulk density of the starting calcite was 1.37 g cm^{-3}; specific surface area of calcite (BET), 0.06 m^2 g^{-1}; pore volume, 1.0×10^{-3} cm^3 g^{-1}; and average pore diameter, 52.3 nm. The 0.2–0.25-mm fraction was used in the study.

The Ca/S ratio was varied by changing the flow rates of liquid fuels and the expenditure of calcite. Experiments were performed at the following initial concentrations of SO_2: 616 (SO), 720 (TO), 900 (TO), and 1450 ppm (TO).

The concentration of combustion products in effluent gases was determined with a Polar analyzer manufactured by Promekopribor OOO, Russia.

Experiments on determining the capacity of calcite and the degree of SO_2 conversion to SO_3 on the catalyst were performed in a reactor with fixed bed and gas moving from top to bottom. The reaction diameter was 23 mm, the calcite charge was 0.8 and 2.0 cm^3, and the catalyst charge, 0.8 cm^3. To provide a uniform distribution of the gas over the reactor cross-section across the calcite layer, porous 5-mm-thick quartz layers with hydraulic resistance exceeding that of the calcite were used, with the calcite layer situated between the plates. The process was performed at temperatures of 600 and 700°C and gas flow rate of 75 L h^{-1} (standard conditions) and the following gas composition at the reactor inlet: volume fraction of SO_2, 520–600 ppm; volume fraction of oxygen, 13.5%; and nitrogen the rest.

The change in the structural characteristics of calcite in the fluidized catalyst bed was determined on passing 83 g h^{-1} of calcite across the catalyst bed at 700°C in the course of 1 h. The catching was performed with a TsN-15 cyclone. Calcite caught in the cyclone was sieved to determine its fraction composition. The content of calcium in the catalyst after the deposition of calcite was determined by the X-ray fluorescence method on the ARL-Advant'x analyzer (Switzerland) with Rh-anode of the X-ray tube.

The texture characteristics of calcite and catalyst were determined on the basis of isotherms of the low-temperature adsorption of nitrogen (ASAP-2400, Micromeritics, United States).

RESULTS AND DISCUSSION

Taking into account that sulfur accumulates to a certain level in oxidation of sulfur-containing organic substances in a fluidized catalyst bed, we performed experiments on determining how the Ca/S ratio affects the variation of the SO_2 concentration at the reactor outlet after the catalyst is saturated with sulfur. This was done with heavy oil burnt in the catalyst bed until the SO_2 concentration ceased to change, which corresponds to the equilibrium state at which the formation rate of sulfates in the catalyst is equal to the rate of their decomposition. The process was performed at a 1.9-fold excess of air, with the expenditure of heavy oil being 260 g h^{-1}.

To calculate and optimize the system in which the burning of liquid fuels occurs with catalytic oxidation of SO_2 and absorption of sulfur oxides by calcite, it is
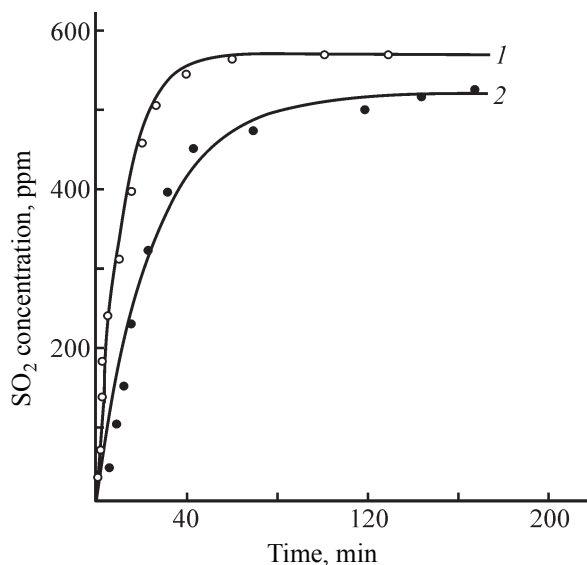
Fig. 2. Time dependence of the SO$_2$ concentration at the outlet of a reactor with fixed bed at different calcite charges at $T = 700°C$. (*1*) Calcite charge (V_{cal}) is 0.8 cm^3, $c_{SO2}{}^0 = 570$ ppm; and (*2*) $V_{cal} = 1.4$ cm^3, $c_{SO2}{}^0 = 520$ ppm. Points, experiment; lines, calculation.
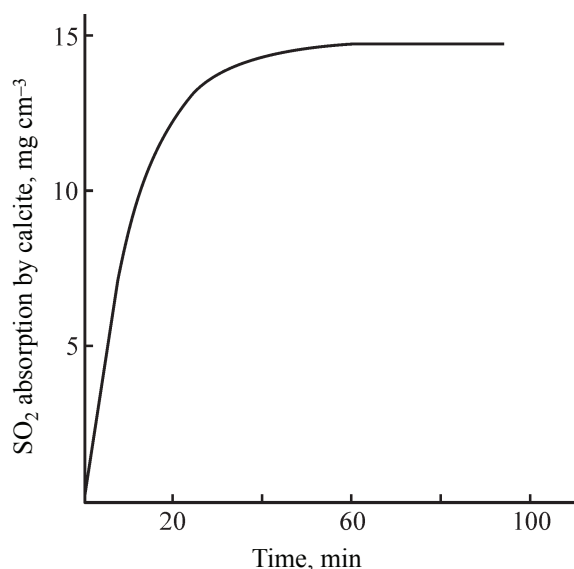

Fig. 3. Dynamics of sulfur accumulation on 1 cm^3 of calcite in a fixed bed at 700°C.

necessary take into account the adsorption properties of calcite and the properties of the catalyst as regards the conversion of SO$_2$ to SO$_3$.

Determination of the capacity of calcite. By the capacity of calcite is understood the ultimate amount of SO$_2$ that can be absorbed (reacted) under the given conditions.

The rate constant of sulfur dioxide absorption by calcite and its capacity were found from the experimental dependence of how the SO$_2$ concentration varies with time by using the equation describing the variation of the SO$_2$ concentration at the exit of the catalyst bed in the following form

$$dc/dt = k_a(c_0 - c), \quad (1)$$
$$\text{at } t = 0 \ c = 0,$$

where c_0 and c are the molar fractions of SO$_2$ at the entry into the layer and its running value at the exit from the layer; t, time; and k_a rate constant of the absorption of sulfur by calcite (s^{-1}). The calculation was based on experimental data on the absorption of SO$_2$ by calcite in a fixed bed at a temperature of 700°C. The experiments were performed at various calcite charges into the rector. The content of oxygen in the flow was 13.5 vol %, and the content of SO$_2$ was varied within the range 520–570 ppm to separate the relaxation curves of sulfur dioxide output. The calculated and experimental data are compared in Fig. 2.

As the volume of calcite in the reactor increases, both the time in which calcite is saturated and the amount of absorbed substance grow. For example, 11.2×10^{-3} g of the substance was absorbed at a calcite charge of 0.8 cm^3 in ~40 min, whereas at 1.4 cm^3, 22.7×10^{-3} g was absorbed in ~90 min. However, the specific capacities $a*$ of the sorbent rather well coincide in both cases, being $(14.2–15.9) \times 10^{-3}$ g cm^{-3}.

Data on the variation of the sulfur dioxide concentration $c_{SO2}(t)$ at the exit from the bed can be used to calculate the absorption dynamics of sulfur dioxide by calcite and its capacity by the following formula

$$a(t) = \frac{V_g c_t M_{SO_2}}{V_{cal}} \int_0^t [c^0 - c_{SO_2}(t)]dt, \quad (2)$$

where, $a(t)$ is the content of sulfur at a given instant of time (mg cm^{-3}); V_g, gas flow rate (cm^3 s^{-1}); c_t number of moles in unit volume of the gas (mol cm^{-3}); M_{SO_2}, molecular weight of sulfur (mg mol^{-1}); and V_{cal}, amount of the adsorbent (cm^3).

The time dependence of the calculated value of $a(t)$ is shown in Fig. 3.

An elemental analysis of calcite demonstrated that the content of sulfur was 14.9×10^{-3} g cm^{-3}, in good agreement with the values obtained in experiments

Table 2. Fraction composition of calcite particles at the reactor outlet

Particle size, mm	0.2–0.25	0.18–0.2	0.1–0.18	0.071–0.1	0.007–0.071	<0.007
Content, wt %	0.05	0.54	3.07	18.77	57.36	20.21

concerned with the absorption kinetics of sulfur oxides on calcite.

The experiment on how the calcite structure changes on passing across the fluidized bed of the catalyst at 700°C demonstrated that the starting particles are disintegrated and the porous structure is changed. The degree of gas cleaning to remove calcite particles was 79.8%. At this degree of gas cleaning, the maximum size of particles caught by the cyclone is 7 μm [20]. The fraction composition of calcite particles at the reactor outlet is presented in Table 2.

Calcite particles had at the reactor outlet the following porous structure characteristics: specific surface area of calcite (BET) 5.74 m^2 g^{-1}, pore volume 14.1 × 10^{-3} cm^3 g^{-1}, and average pore diameter 9.5 nm. The specific surface area sharply increases (by a factor of 13) as compared with calcite thermally treated in a fixed bed. Accordingly, the pore volume increases, with the average pore size changing only slightly. This effect is due both to the decrease in the size of calcite particles and to the high-intensity thermal impact in the fluidized bed in which the heat-transfer coefficient is several times that in the case of a slow heating in a fixed bed.

The capacity of calcite under the conditions of a fluidized catalyst bed can be evaluated on the basis of results on the absorption in a stationary bed on the assumption that the whole accessible area is involved in the absorption process. In this case, the calcite capacity in a fluidized bed is given by

$$\alpha_{flb} = \alpha^* S_{flb}/S_{fxb} = 0.194 \text{ g SO}_2/\text{cm}^3,$$

where $S_{flb} = 5.74$ m^2 g^{-1} is the specific surface area of calcite in the fluidized bed at 700°C, $S_{fxb} = 0.44$ m^2 g^{-1} is the specific surface area of calcite in the fixed bed at 700°C.

Thus, the potential capacity of calcite in a fluidized bed exceeds by an order of magnitude the capacity of calcite in a fixed bed. This occurs as a result of the sharp increase in the specific surface area due to the fragmentation of particles, change in the internal structure of calcite, and deposition of calcite on the catalyst surface.

Study of the interaction of SO$_2$ with the catalyst in fixed and fluidized catalyst beds. The industrial process of the catalytic oxidation of SO$_2$ to SO$_3$ is of the large-tonnage kind. Kinetic models have been developed for industrially used catalysts. For example, several tens of kinetic equations have been suggested in the literature for simulation and optimization of the reaction course on V–Ti catalysts [21]. The ShchKZ-1 catalyst is of the aluminum–copper–chromium oxide type. There are no data for this catalyst on the conversion of SO$_2$ to SO$_3$ at various temperatures, and no kinetic models describing the conversion of SO$_2$ to SO$_3$ have been developed. It is known [22] that the activity of the chromium oxide catalyst exceeds that of the iron oxide and copper oxide catalysts and is lower than that of the vanadium catalyst. It has been noted that the degree of SO$_2$ conversion to SO$_3$ upon a prolonged keeping at a temperature of 587°C is 83%. This value is correlated with the results of experiments on burning of sulfur-containing fuels in the presence of ShchKZ-1 in a fluidized bed. As shown below, a degree of SO$_2$ conversion to SO$_3$ of about 78–80% is reached in a fluidized bed of ShchKZ-1 catalyst at a temperature of 585°C. Taking into account that copper oxide contained in ShchKZ-1 catalyst has a lower activity as compared with the chromium oxide catalyst [22], we can conclude the degree of SO$_2$ conversion to SO$_3$, obtained with ShchKZ-1 catalyst, is provided by
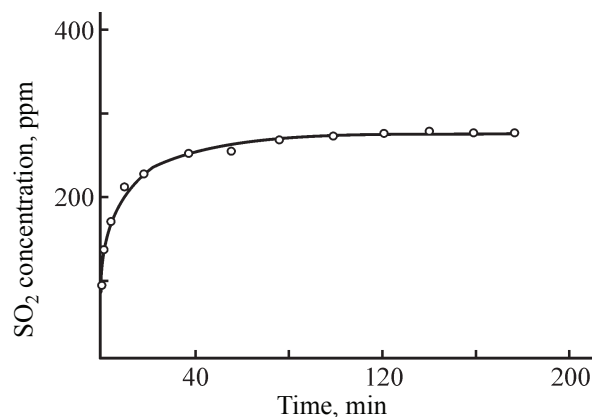

Fig. 4. Variation with time of the SO$_2$ concentration at the exit from the fixed catalyst bed at 700°C. The SO$_2$ concentration at the reactor inlet was 570 ppm, and the oxygen content was 13.5%.
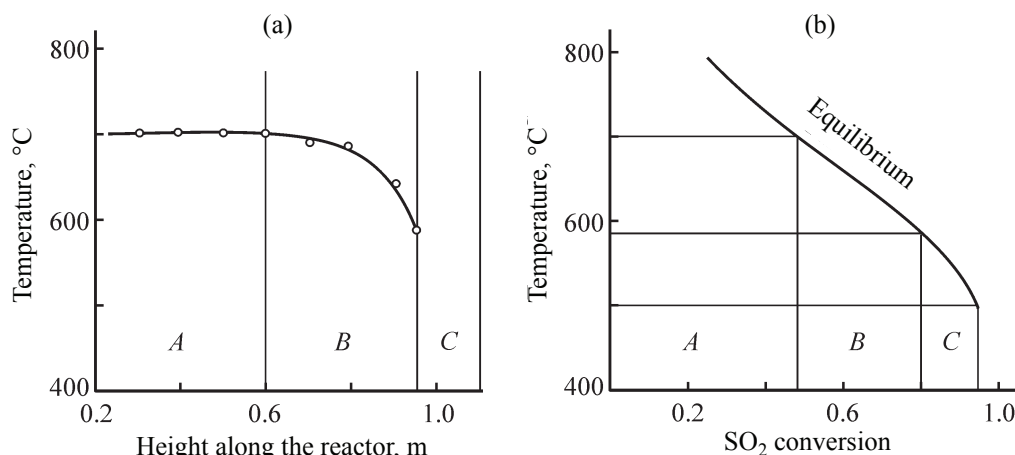
Fig. 5. (a) Temperature profile in the reactor on placing the heat exchanger in the upper part of the reactor and (b) dependence of the equilibrium conversion of SO_2 into SO_3. (*A*) Oil burning zone; (*B*) zone of additional oxidation of SO_2, with the temperature at the exit from the fluidized catalyst bed of 585°C (transition zone); (*C*) zone of heat removal by the heat exchanger.

chromium oxide. At a temperature of about 500°C [22], the degree of SO_2 conversion to SO_3 on a chromium oxide catalyst may exceed 90%. However, chromium oxide is not used industrially to perform the oxidation of SO_2 to SO_3 because sulfates are formed at a temperature of about 500°C.

Under the conditions of SO_2 oxidation to SO_3 at a simultaneous oxidation of organic substances in a fluidized bed, e.g., with IK-12-2 industrial aluminum–copper–chromium oxide catalyst, no significant sulfation of the catalyst occurs, and the content of sulfates remains upon a prolonged operation of the catalyst about 0.65% in terms of sulfur [18]. One of specific features of the fluidized bed is the circulation of particles over the whole layer. Therefore, it would be expected that lowering the temperature to 500°C in the upper part of the fluidized bed will not lead to a significant sulfation of the catalyst because of its subsequent penetration into the fuel-burning zone with temperature of 700°C.
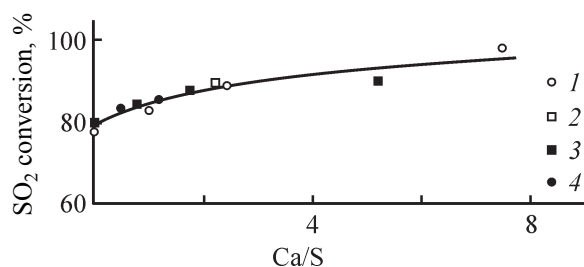

Fig. 6. SO_2 conversion at the outlet of a reactor with fluidized catalyst bed vs. the Ca/S rtio in burning of liquid fuels with varied content of sulfur, $T_{out} = 585$°C. Initial SO_2 concentration c_0 (ppm): (*1*) 616 (SO), (*2*) 720 (HO), (*3*) 900 (HO), (*4*) 1450 (HO). Points, experiment; lines, calculation.

When the reaction mixture is delivered to the catalyst, the latter operates during the initial interval of time in the nonstationary mode, i.e., the content of sulfur dioxide, c_{SO_2}, at the reactor outlet varies. After a certain time, c_{SO_2} reaches its steady-state value, which corresponds in the given case to a 48% SO_2 conversion to SO_3 (Fig. 4).

To more effectively catch sulfur oxides by calcite, it is necessary to raise the conversion of SO_2 to SO_3, which has a higher reactivity. This can be done by lowering the temperature at the exit from the catalyst bed. To lower the temperature, we organized a nonisothermal profile by placing the heat exchanger in the upper part of the reactor, with the temperature of the gas at the reactor outlet being 585°C (Fig. 5a).

The equilibrium SO_2 concentration x_{eq} can be estimated by using the formula reported in [22]:

$$x_{eq} = \frac{K_{eq}}{K_{eq} + \sqrt{\dfrac{100 - 0.5ax_{eq}}{P(b - 0.5ax_{eq})}}}, \qquad (3)$$

where K_{eq} is the equilibrium constant of the reaction of SO_2 oxidation to SO_3 by oxygen; a, content of SO_2 in the starting gas mixture (vol %); b, content of oxygen in the starting gas mixture (vol %); and P, total gas pressure (atm).

The calculation was performed by the trial-and-error method. The equilibrium constant is given by

$$K_{eq} = \left(\frac{c_{SO_3}}{c_{O_2}^{0.5} c_{SO_2}} \right)_{eq}. \qquad (4)$$

The dependence of the equilibrium constant K_{eq} of the reaction was determined by the formula [22]

$$\log K_{eq} = 4905/T - 4.6455.$$

The temperature dependence of the equilibrium conversion, calculated up to 500°C, is shown in Fig. 5b.

When calcite is delivered to the catalyst bed, a decrease in the SO_2 concentration is observed and, accordingly, an increase in the apparent conversion of SO_2, which depends on the Ca/S ratio. Figure 6 shows the results obtained in burning of SO and TO in a boiling bed of ShchKZ-1 catalyst in the absence of calcite and with batching of calcite at various Ca/S ratios.

The total degree of carbon dioxide conversion X will be the sum of conversions via transformation on the catalyst (X_C) and calcite (X_{Ca}):

$$X = X_C + X_{Ca}. \tag{5}$$

Thus, when calcite is batched into the reactor with a fluidized catalyst bed, the apparent conversion at the reactor outlet with a pseudofluidized bed of ShchKZ-1 catalyst additional grows, with this conversion dependent on the Ca/S molar ratio. At Ca/S = 4.1, which corresponds to the capacity of calcite used in the study, a conversion of SO_2 of about 90% is reached. This indicates that the absorption capacity of calcite is not fully utilized. As Ca/S ratio is raised to 7.6, the conversion becomes as large as 99%.

CONCLUSIONS

The catalytic burning of sulfurous and heavy high-sulfur oil in a fluidized bed of an aluminum–copper–chromium catalyst with addition of a solid agent binding sulfur oxides, calcite, was studied. A mathematical model of the process was suggested on the basis of the experimental results obtained in the study. In addition, it was shown that the process is the most effective for the nonisothermal temperature profile, with the temperature decreasing downstream from 700 to 500°C.

With the process performed in the nonisothermal temperature mode and calcite delivered into the reactor, the total degree of conversion reaches a value of 99% (at a Ca/S molar ratio of 7.6).

The experimental data on the variation of the concentration of sulfur at the exit from a fixed bed of

calcite were used to calculate its specific capacity to be $(14.2–15.9) \times 10^{-3}$ g cm^{-3}. It was shown that the potential capacity of calcite in the fluidized bed is 14.2×10^{-2} g SO_2/g calcite, which exceeds by an order of magnitude the value obtained for the fixed bed. This difference is due to the sharp increase in the specific surface area of calcite in the fluidized bed (by a factor of 13) because of the particle fragmentation as compared with calcite thermally treated in a fixed bed.

Thus, the burning of sulfur-containing liquid fuels in a nonisothermal fluidized bed of a catalyst makes it possible to combine the full oxidation of the fuel at 700°C with the effective additional transformation of SO_2 at 500°C at the reactor outlet and is a promising way to industrial implementation of the process. The data obtained can be used when designing industrial installations for ecologically safe burning of sulfur-containing fuels, including wastes from oil extraction and oil processing.

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CONFLICT OF INTERESTS

The authors maintain that there is no conflict of interest to be disclosed in the present communication.

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