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Kinetic Model of the Reaction of Methanol with Hydrogen Sulfide

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Abstract—The kinetics of the reaction of methanol with hydrogen sulfide in the presence of an IKT-31 catalyst was experimentally studied. The experiments were performed in a fixed-bed flow reactor under the following conditions: $T = 598$–653 K, $P = 0.1$–1.0 MPa, and $P^0_{H_2S}/P^0_{Me} = 0.4$–$15.0$. Rate equations were derived which describe the rates of formation of methanethiol as the main product and dimethyl sulfide and dimethyl ether as by-products. The rate constants and activation energies were found by the mathematical treatment of experimental data. The model proposed can be used for reactor design.

INTRODUCTION

The heterogeneous catalytic reaction of hydrogen sulfide with methanol is of importance because it forms the basis for the commercial production of methanethiol, which is used in the synthesis of methionine (a pharmaceutical and a supplement to food for birds). In the presence of catalysts used in this process (alumina modified with an alkali and tungsten compounds), methanol thiolation with the formation of methanethiol predominates under certain conditions [1]. Mashkin and coauthors [2, 3] studied the kinetics of the interaction of methanol with H_2S at atmospheric pressure in a gradientless reactor at $T = 593$–673 K in the presence of an alumina–potassium–tungsten catalyst. Kinetic models were constructed with consideration for surface heterogeneity. The industrial synthesis of methanethiol is performed at a pressure of ~1 MPa [4]. The effect of pressure on the kinetics of the reaction of methanol with H_2S was not previously studied.

The aim of this work was to study experimentally the kinetic features of the interaction of methanol with H_2S at elevated pressures in the presence of an IKT-31 commercial catalyst and to construct a kinetic model.

EXPERIMENTAL

The study was performed in a fixed-bed flow reactor with a fine-grained catalyst (particle size of 0.25–0.5 mm). A mixture of hydrogen sulfide with helium arrived at a thermostatted bubbler sending methanol through a pressure regulator; next, the saturated gas was directed to the reactor packed with the catalyst. The reactor diameter was 12 mm, and the height of a catalyst bed was 200 mm. In the experiments, the flow rate, temperature, and total pressure were varied within the ranges 0.2–1.0 cm^3/s, 598–653 K, and 0.1–1.9 MPa, respectively. The partial pressure of methanol in the feed gas varied within the range 0.004–0.015 MPa, and the ratio between hydrogen sulfide and methanol was 0.4–15.0.

The conversion of methanol under the specified conditions changed from 25 to 97%. The feed gas and the reaction products were analyzed on a Tsvet-500 chromatograph (a 2 m × 3 mm column packed with a mixture of Porapak Q and Porapak R (1 : 1); helium was a carrier gas). The error of analysis was ±5 rel %.

RESULTS AND DISCUSSION

Methanethiol and water were the main reaction products. Dimethyl sulfide (DMS) and dimethyl ether (DME) were detected in small amounts, as well as CH_4, CO, and CO_2 traces. Figures 1 and 2 demonstrate typical experimental data, which illustrate the yields of reaction products at 613–653 K and pressures of 0.1 and 1 MPa.

Figure 1 graphically illustrates the distribution of sulfur, which was introduced into the reactor with a flow of hydrogen sulfide, in the products as the dependence of the yields of methanethiol (Y^1_{MT}) and DMS (Y^1_{DMS}) on the conversion of hydrogen sulfide (X_{H_2S}). Data shown in these graphs were obtained in an excess of hydrogen sulfide ($P^0_{H_2S}/P^0_{Me} = 1.5$–$1.7$, where $P^0_{H_2S}$ and P^0_{Me} are the partial pressures of H_2S and methanol (Me) at the reactor inlet, respectively). The values of $Y^{(1)}_{MT}$ and $Y^{(1)}_{DMS}$ were determined as molar flows at the

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[1] V.N. Yakovleva and L.G. Sakhal'tueva participated in the experiments.

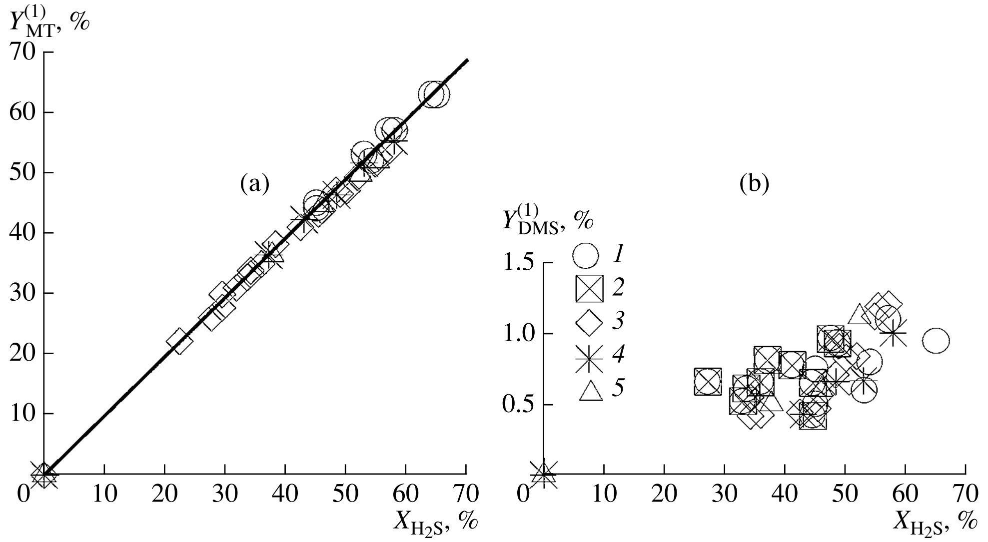

Fig. 1. Dependence of the yields ($Y^{(1)}$) of (a) methanethiol and (b) DMS on the conversion of hydrogen sulfide (X_{H_2S}) at P = (*1*) 0.1 and (*2*–*5*) 1.0 MPa and T = (*1*, *3*) 633, (*2*) 613, (*4*) 648, and (*5*) 653 K. $P^0_{H_2S}/P^0_{Me}$ = 1.7–1.8.

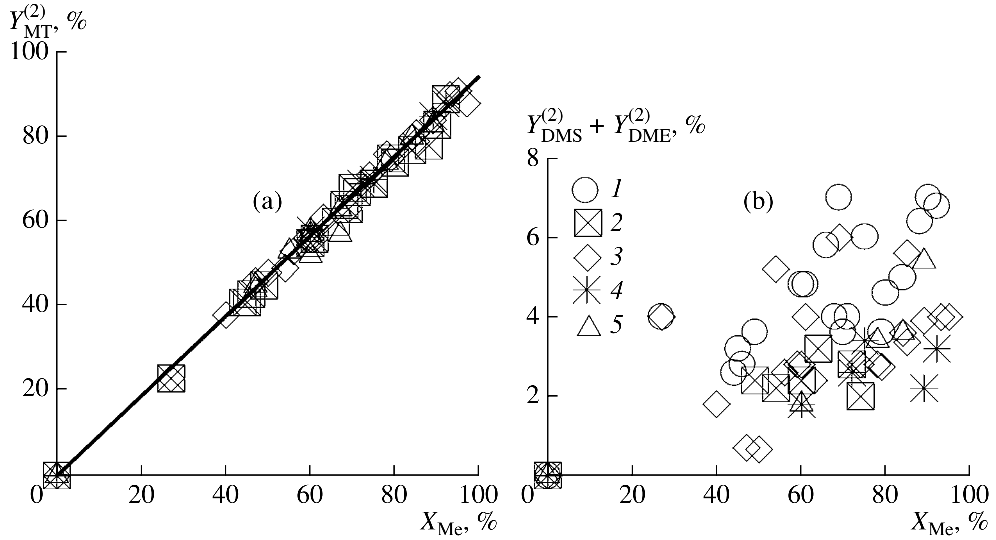

Fig. 2. Dependence of (a) the yield of methanethiol ($Y^{(2)}$) and (b) the doubled sum of the yields of DMS and DME on the conversion of methanol (X_{Me}) at P = (*1*) 0.1 and (*2*–*5*) 1.0 MPa and T = (*1*, *3*) 633, (*2*) 613, (*4*) 648, and (*5*) 653 K. $P^0_{H_2S}/P^0_{Me}$ = 1.7–1.8.

reactor outlet multiplied by 100 and divided by the molar flow of hydrogen sulfide at the reactor inlet.

The rectilinear dependence of the yield of methanethiol on X_{H_2S} suggests that methanethiol is the main final product, which is formed in the reaction

$$CH_3OH + H_2S \longrightarrow CH_3SH + H_2O. \qquad (I)$$

The slope of the straight line in Fig. 1a is equal to 0.97; it characterizes the selectivity of the reaction for hydrogen sulfide, which is determined from the equation

$$S^{(1)}_{MT} = \frac{n_{MT}(\tau)}{n^0_{H_2S} - n_{H_2S}(\tau)} = \text{const} \qquad (1)$$

at all contact times (τ),

where n_{MT} and n_{H_2S} are the molar flows of methanethiol and H_2S, respectively, at the reactor outlet, and $n^0_{H_2S}$

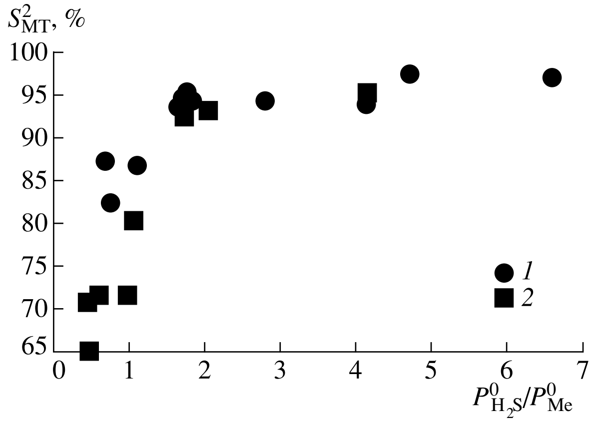

Fig. 3. Dependence of the selectivity for methanethiol (S^2_{MT}) on the ratio of the partial pressures of hydrogen sulfide and methanol in the initial mixture. P = (*1*) 1.0 and (*2*) 0.1 MPa. T = 633 K.

is the molar flow of hydrogen sulfide at the reactor inlet. It can be seen in Fig. 1a that, within the limits of experimental error, $S^{(1)}_{\mathrm{MT}}$ is independent of hydrogen sulfide conversion, as well as of temperature and pressure.

The yield of another sulfur-containing product, DMS (Fig. 1b), is at most 2% of converted hydrogen sulfide. DMS can be formed in the following reactions [3]:

$$\mathrm{CH_3SH + CH_3OH \longrightarrow (CH_3)_2S + H_2O}, \qquad \text{(II)}$$

$$\mathrm{2CH_3SH \rightleftharpoons (CH_3)_2S + H_2S}, \qquad \text{(III)}$$

$$\mathrm{2CH_3OH + H_2S \longrightarrow (CH_3)_2S + 2H_2O}. \qquad \text{(IV)}$$

Reactions (II) and (III) imply the successive conversion of methanethiol into DMS, whereas reaction (IV) implies DMS formation through a parallel reaction path. Because the yield of DMS is low and falls within the limits of an experimental imbalance of sulfur, it is evident that with the use of these experimental data we cannot evaluate the predominant contribution of any one of the above reactions to the formation of DMS. At the same time, Mashkin *et al.* [3] observed a decrease in the selectivity for methanethiol with increasing conversion of hydrogen sulfide.

Figure 2 demonstrates analogous balance relationships obtained under the same conditions as the data in Fig. 1. The ordinate in Fig. 2a is the yield of methanethiol normalized to the molar flow of methanol at the reactor inlet ($Y^{(2)}_{\mathrm{MT}}$), which is plotted as a function of methanol conversion. The slope of the straight line in Fig. 2a is equal to 0.94; it characterizes the selectivity of methanol consumption for main reaction (I):

$$S^{(2)}_{\mathrm{MT}} = \frac{n_{\mathrm{MT}}(\tau)}{n^0_{\mathrm{Me}} - n_{\mathrm{Me}}(\tau)} = \text{const} \quad \text{at all contact times.} \qquad (2)$$

The other 0.06 parts of methanol were partially consumed in reaction (IV) and in the formation of DME by the reaction

$$\mathrm{2CH_3OH \rightleftharpoons (CH_3)_2O + H_2O}. \qquad \text{(V)}$$

Figure 2b represents the doubled sum of the yields of DMS and DME normalized to the molar flow of methanol at the reactor inlet as a function of the degree of methanol conversion. The scatter of data is large; however, it falls within the limits of an experimental imbalance with respect to the methyl group.

The degrees of conversion of methanol and hydrogen sulfide were determined from the equations

$$X_{\mathrm{Me}} = (n^0_{\mathrm{Me}} - n_{\mathrm{Me}}) \times 100/n^0_{\mathrm{Me}}, \qquad (3)$$

$$X_{\mathrm{H_2S}} = (n^0_{\mathrm{H_2S}} - n_{\mathrm{H_2S}}) \times 100/n^0_{\mathrm{H_2S}}. \qquad (4)$$

As noted above, the relationships represented in Figs. 1 and 2 are valid for all the pressures and temperatures tested with an excess of H_2S in the reaction mixture ($P^0_{\mathrm{H_2S}}/P^0_{\mathrm{Me}} > 1$). At the same time, in an excess of methanol (i.e., at $P^0_{\mathrm{H_2S}}/P^0_{\mathrm{Me}} < 1$), $S^{(2)}_{\mathrm{MT}}$ essentially depends on the $P^0_{\mathrm{H_2S}}/P^0_{\mathrm{Me}}$ ratio (Fig. 3). In Fig. 3, the values of $S^{(2)}_{\mathrm{MT}}$ obtained experimentally by varying the values of $P^0_{\mathrm{H_2S}}/P^0_{\mathrm{Me}}$ over a wide range at T = 633 K and P = 0.1 or 1.0 MPa are plotted as ordinates. Data shown in Fig. 3 indicate that, in an excess of methanol, the ratio of the yield of methanethiol to the amount of reacted methanol considerably decreased.

Figure 4 illustrates the dynamics of the process as the dependence of X_{Me}, $X_{\mathrm{H_2S}}$, and $Y^{(2)}_{\mathrm{MT}}$ on contact time (τ) at two pressures (0.1 and 1.0 MPa) and T = 633 K. The contact time was determined as the ratio of the catalyst weight to the total molar flow of a gas at the reactor inlet.

It can be seen in the graphs that, at the initial portions of the curves, the degrees of conversion of the initial reactants increased practically linearly with contact time up to approximately 20–40% conversions. Then, a

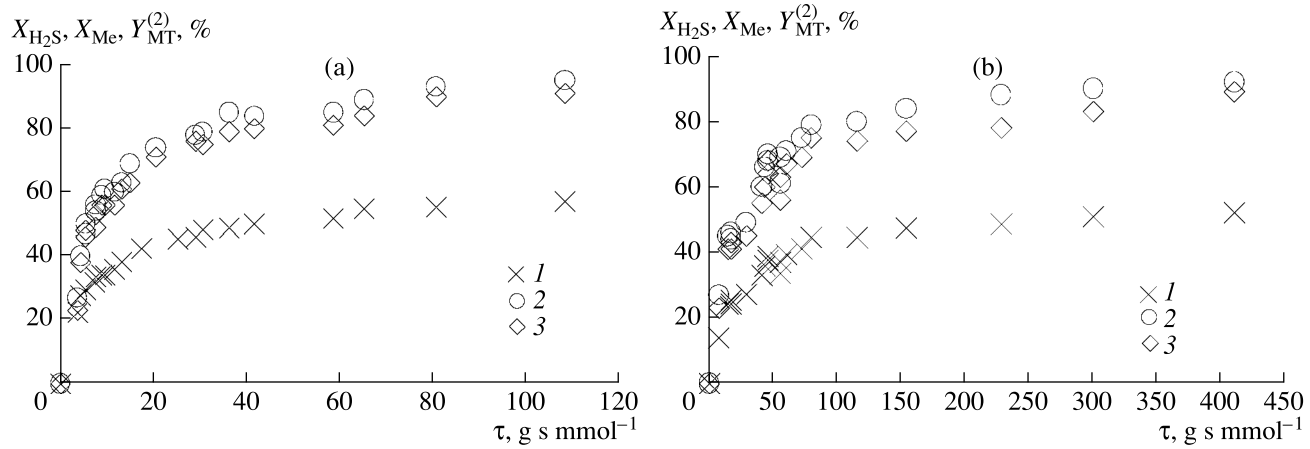

Fig. 4. Dependence of the conversions of methanol and hydrogen sulfide and the yield of methanethiol on contact time. $T = 633$ K, $P^0_{H_2S}/P^0_{Me} = 1.7–1.8$, P = (a) 1.0 and (b) 0.1 MPA. (*1*) X_{H_2S}, (*2*) X_{Me}, and (*3*) $Y^{(2)}_{MT}$.

dramatic decrease in the gradients of growth of both X_{Me} and X_{H_2S} was observed. These regularities allowed us to assume that the process is strongly inhibited by reaction products.

A comparison of Figs. 4a and 4b demonstrates that the rate of reaction increases with pressure. The temperature dependence of the initial rates of methanethiol formation ($w_0 = \Delta Y_{MT}/\Delta\tau$) at $\tau \longrightarrow 0$, which is represented in Fig. 5, allowed us to obtain a primary estimate of the activation energy of the main reaction. It is equal to ~65 kJ/mol.

To evaluate the displacement from an equilibrium state in our kinetic experiments, we performed calculations by the minimization of the Gibbs free energy function in accordance with a published procedure [5]. Table 1 summarizes the results of the calculations at $T = 633$ K (depending on the molar numbers of H_2S and methanol in the initial mixture). In this case, the effect of the total pressure on the equilibrium composition of the mixture is insignificant because the number of moles in equilibrium changed insignificantly, as compared with the initial value.

Data in Table 1 demonstrate the thermodynamic probability of sulfur distribution between two sulfur-containing products and the probability of methyl-group distribution between the main product and by-products. The equilibrium yield of DMS is reasonably high, and it decreases with increasing excess of hydrogen sulfide. At the same time, the equilibrium yield of methanethiol increased with increasing excess of H_2S. Note that DME was completely absent from the equilibrium reaction mixture. DME, which is formed in reaction (V), can be consumed in the reactions

$$(CH_3)_2O + CH_3SH \rightleftharpoons (CH_3)_2S + CH_3OH, \qquad (VI)$$

$$(CH_3)_2O + H_2S \rightleftharpoons CH_3SH + CH_3OH. \qquad (VII)$$

These reactions are thermodynamically possible; they were observed on other catalysts [3]. However, the rates of these reactions on the given catalyst are insignificant, and we cannot observe them.

Based on the experimental data, we chose one of the probable sets of independent reactions, which included reactions (I), (IV), and (V), for constructing a kinetic model. These reactions predict parallel reaction paths for the formation of not only methanethiol and DME but also DMS. This approximation can be valid under the conditions of our experiments because the yields of the DMS and DME products are insignificant as com-

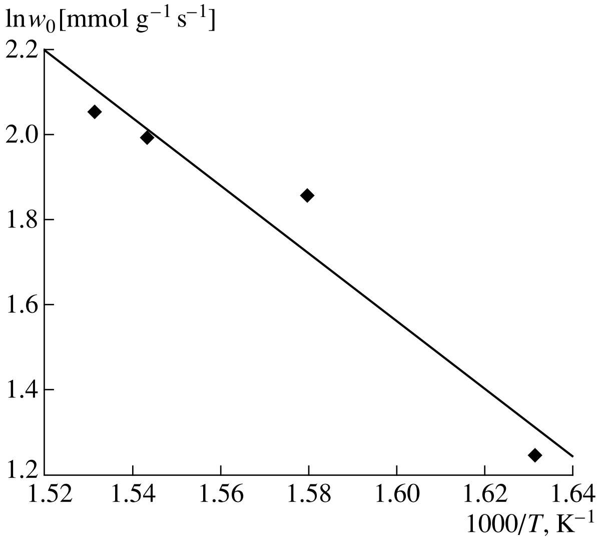

Fig. 5. Dependence of the initial rate of methanethiol formation on temperature in the ln $w_0(\tau \longrightarrow 0) – 1000/T$ coordinates.

Table 1. Equilibrium compositions (mol) of the reaction mixture depending on $n^0_{H_2S}/n^0_{Me}$

Component	$n^0_{H_2S}/n^0_{Me}$				
	0.5/1.0 $n^0_{Me} = 1.0$	1.0/1.0	2.0/1.0	3.0/1.0	4.0/1.0
CH_3OH	0.010	0.000	0.000	0.000	0.000
H_2S	0.000	0.351	1.259	2.211	3.180
CH_3SH	0.010	0.296	0.482	0.578	0.640
$(CH_3)_2S$	0.490	0.352	0.259	0.211	0.180
$(CH_3)_2O$	0.000	0.000	0.000	0.000	0.000
H_2O	0.990	1.000	1.000	1.000	1.000

Note: $T = 633$ K, $P = 0.1$–1.0 MPa.

pared with the yield of methanethiol. Moreover, we cannot give an accurate kinetic description of reactions (I), (III), and (VI), which result in the further conversion of methanethiol, because of the large scatter of data on the yields of DMS and DME and the absence of maxima from the curves of the yield of methanethiol plotted as a function of the conversion of initial reactants.

The catalytic reaction of methanol with hydrogen sulfide was studied in detail with the use of adsorption techniques, IR spectroscopy, and NMR spectroscopy [1–3]. It was assumed that the reaction occurs via the main steps considered below. In the interaction of methanol with a catalyst, the dissociative chemisorption of methanol occurs with the methoxylation of the surface:

$$\begin{gathered} CH_3OH + [\theta_1] \rightleftharpoons [\theta_2] + H_2O, \\ w_1 = k_1 P_{Me}[\theta_1] - k_{-1} P_{H_2O}[\theta_2]. \end{gathered} \tag{VIII}$$

Here, $[\theta_1]$ are the initial active sites of the surface, and $[\theta_2]$ are the sites covered with CH_3O. The resulting methoxy groups react with activated H_2S to form methanethiol:

$$\begin{gathered} H_2S + [\theta_2] \longrightarrow [\theta_1] + CH_3SH, \\ w_2 = k_2 P_{H_2S}[\theta_2]. \end{gathered} \tag{IX}$$

Next, methanol and methanethiol from a gas phase or activated at other sites also react with methoxide groups to result in the formation of DMS and DME:

$$\begin{gathered} CH_3SH + [\theta_2] \longrightarrow [\theta_1] + (CH_3)_2S, \\ w_3 = k_3 P_{MT}[\theta_2], \end{gathered} \tag{X}$$

$$\begin{gathered} CH_3OH + [\theta_2] \longrightarrow [\theta_1] + (CH_3)_2O, \\ w_4 = k_4 P_{Me}[\theta_2]. \end{gathered} \tag{XI}$$

The water formed in this process is adsorbed at the $[\theta_1]$ sites to form the $[\theta_3]$ sites covered with water:

$$\begin{gathered} H_2O + [\theta_1] \rightleftharpoons [\theta_3], \\ w_5 = k_5 P_{H_2O}[\theta_1] - k_{-5}[\theta_3]. \end{gathered} \tag{XII}$$

Steps (IX)–(XI) are slow, as compared with steps (VIII) and (XII), which occur in equilibrium.

The above reaction sequence implies that methanethiol is an intermediate product and participates in the formation of DMS. As noted above, thermodynamic calculations and experimental data on the thiolation of methanol (including experiments with the addition of water to the initial mixture) on other catalysts [3] are consistent with this mechanism. However, because we did not observe a decrease in the yield of methanethiol to within the experimental error with increasing methanol conversion, we eliminated step (X) of the successive conversion of methanethiol for deriving reaction rate equations. With consideration for the relation

$$[\theta_1] + [\theta_2] + [\theta_3] = 1 \tag{5}$$

and on the assumption that steps (VIII) and (XII) are in equilibrium, we can write the following reaction rate equations for the other steps:

$$w_{MT} = \frac{k_{MT} K_1 P_{H_2S} P_{Me}}{P_{H_2O} + K_1 P_{Me} + K_5 P^2_{H_2O}}, \tag{6}$$

$$w_{DME} = \frac{k_{DME} K_1 P^2_{Me}}{P_{H_2O} + K_1 P_{Me} + K_5 P^2_{H_2O}}. \tag{7}$$

In Eqs. (6) and (7), $k_{MT} = k_2$, $k_{DME} = k_4$, $K_1 = k_1/k_{-1}$, and $K_5 = k_5/k_{-5}$.

The rate equation for the formation of DMS was chosen based on purely phenomenological considerations on the assumption that overall reaction (IV) is responsible for the formation of DMS:

$$w_{DMS} = \frac{k_{DMS} K_1 P_{H_2S} P^2_{Me}}{P_{H_2O} + K_1 P_{Me} + K_5 P^2_{H_2O}}. \tag{8}$$

In principle, taking into account the large scatter of data and low DMS concentrations (within the limits of an experimental imbalance of sulfur), we could choose any other equation or completely ignore the formation of DMS. At the same time, because the kinetic model is oriented to reactor design calculations, it should reflect the possibility of formation of trace by-product impurities.

Assuming plug flow in the experimental fixed-bed flow reactor, we represent the mathematical model of the experimental reactor as the set of differential equations

$$\frac{d\mathbf{y}}{d\tau} = \mathbf{Z}^T\mathbf{W}, \quad \tau = 0, \quad \mathbf{y} = \mathbf{y}^0, \tag{9}$$

where $\mathbf{y} = [y_{Me}, y_{H_2S}, y_{MT}, y_{H_2O}, y_{DMS}, y_{DME}]^T$ is the vector of the mole fractions of components at the reactor outlet and $\mathbf{y}^0$ is the same at the reactor inlet;

$\mathbf{W} = [w_{MT}, w_{DMS}, w_{DME}]^T$ is the vector of the rates of reactions (I), (IV), and (V), mmol g^{-1} s^{-1};

$\mathbf{Z}$ is the matrix of the stoichiometric coefficients of the above reactions.

The contact time was determined as

$$\tau = \frac{q_{Cat}}{N^0}, \text{ g s mmol}^{-1}, \tag{10}$$

where q_{Cat} is the amount of the catalyst in the reactor, g; N^0 is the total molar flow of reactants at the reactor inlet, mmol/s.

The set of Eqs. (9) was used for the identification of unknown model parameters. The identification was performed using an explicit integral method followed by the reliability analysis of found parameters [6]. Initially, the results of all isothermal experiments at T = 633 K were processed, which were obtained at various total pressures and $P^0_{H_2S}/P^0_{Me}$ ratios. As a result, the numerical values of k_{MT}, k_{DME}, k_{DMS}, K_1, and K_5 at T = 633 K were found. Next, all the data obtained at temperatures of 598, 613, 648, and 653 K were processed together with the found parameters. The activation energies were found from the relations

$$k_i(T) = k_i(633)\exp\left(-\frac{E_i}{R}\left(\frac{1}{T} - \frac{1}{633}\right)\right), \tag{11}$$

where i = MT, DMS, and DME. Analogously, the temperature coefficients of the denominator constants K_1 and K_5 were found from the equations

$$K_i(T) = K_i(633)\exp\left(-\frac{Q_{K_i}}{R}\left(\frac{1}{T} - \frac{1}{633}\right)\right), \quad i = 1, 5. \tag{12}$$

Table 2 summarizes the numerical values and confidence intervals of the constants. The parameters k_{MT}, K_1, K_5, E_{MT}, Q_{K_1}, and Q_{K_5}, which are responsible for the description of the rate of formation of the main product, were determined with a good accuracy. At the same time, note that the accuracy of the determination of k_{DMS}, k_{DME}, E_{DMS}, and E_{DME}, which are responsible for the yields of DMS and DME, was low. These values were determined with a wide confidence interval, and they can be considered insignificant in terms of statistical characteristics. At the same time, the elimination of reactions that result in the formation of by-products from the model violates the meaning of the model because in this case we cannot describe the selectivity of the reaction for the target product. On this basis, the values of the above poorly determined constants presented in Table 2 should be taken for performing practical calculations.

Table 2. Numerical values and confidence intervals of the parameters of the kinetic model (at a 95% confidence level)

Parameter	Value at T = 633 K
k_{MT}, mmol g^{-1} h^{-1} MPa^{-1}	3.78 ± 0.11
k_{DME}, mmol g^{-1} h^{-1} MPa^{-1}	0.14*
k_{DMS}, mmol g^{-1} h^{-1} MPa^{-2}	0.68*
K_1	1618.5 ± 562.8
K_5, MPa^{-1}	2904.7 ± 504.7
E_{MT}, kJ/mol	69.75 ± 9.76
E_{DME}, kJ/mol	67.02*
E_{DMS}, kJ/mol	0.53*
Q_{K_1}, kJ/mol	3.04 ± 1.06
Q_{K_5}, kJ/mol	–19.49 ± 3.39

* Wide confidence intervals.

The average relative error of the description of experimental data on the main components (CH_3OH, H_2S, methanethiol, and H_2O) was 16.8%. The accuracy of description for DMS and DME was much lower and averaged 50–100%. Taking into account low concentrations of the above components and the large scatter of data, we consider this result acceptable.

An analysis of the kinetic model with consideration for the constants given in Table 2 demonstrated that the formation of methanethiol was of first order with respect to H_2S and did not depend on the concentrations of methanol and water at short contact times, when the partial pressure of water was still insignificant as compared with the partial pressure of methanol. Next, as water accumulated and the partial pressure of methanol decreased, the reaction was dramatically inhibited by water because of the blocking of active centers (step (XII)). The temperature coefficient Q_{K_5} was negative; consequently, the inhibition by water decreased with temperature. At the same time, the equilibrium constant K_1 of step (VIII) weakly depended on temperature.

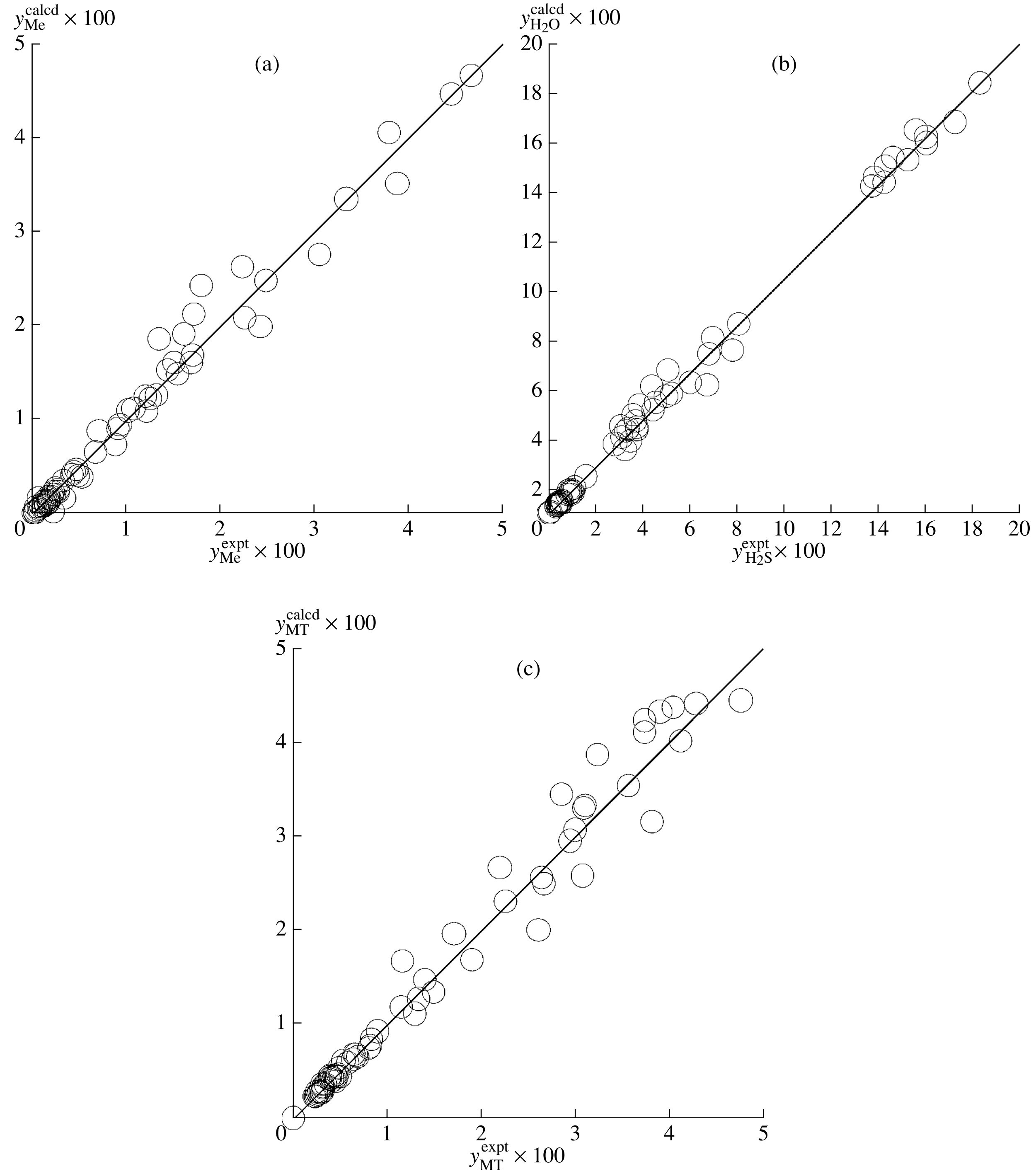

Fig. 6. Comparison between experimental and calculated data for (a) methanol, (b) methanethiol, and (c) hydrogen sulfide. T = 633 K; P = 0.18–1.0 MPa; $P^0_{H_2S}/P^0_{Me}$ = 0.66–1.8.

Figure 6 illustrates experimental and calculated data (at $T = 633$ K, $P = 0.18$–1.0 MPa, and $P^0_{H_2S}/P^0_{Me} = 0.66$–1.8), which suggest that the kinetic model provides satisfactory agreement between them. The deviation between experimental and calculated data is of a random character. Thus, the kinetic model is consistent with the postulated reaction scheme, and it can be recommended for performing practical reactor design calculations in the test temperature region at both atmospheric and elevated pressures. Both the experimental data and the kinetic model suggest that the total pressure in the system affects only the rate of reaction but has no effect on the selectivity. The reaction temperature has an analogous effect. To obtain a maximum selectivity for the target product (methanethiol), we recommend performing the process in a 1.5- to 1.8-fold excess of hydrogen sulfide with respect to the stoichiometry of reaction (I).

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