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Mechanism and Kinetic Model of Heterogeneous Catalystic Process of Demercaptanization of Oil

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ABSTRACT

New heterogeneous and catalytic systems were obtained and their activities were further studied to ensure process of a demercaptanization (mercaptan removal) of oil and oil products. Kinetic regularities of a process were researched. The kinetic model and the mechanism of a demercaptanization of an ethyl mercaptan as a model reaction with participation of organic ligand catalyst deposited on clinoptilolite So-Fe, Mn-Fe is suggested by this study.

Introduction

Mercaptan compounds having the composition of oil and oil products have negative impact on the quality of petroleum products and are the cause of the equipment corrosion used during their production, transportation and processing, which eventually leads to their failure and create environmental problems. Despite the fact that there have been many ways developed to purge oil and oil products from other sulfur compounds, the mercaptan sweetening has been studied marginally. Mercaptan sweetening is currently supplied mainly by classical chemical methods, i.e. using the alkaline and acidic reagents and is conducted by neutralization method. However, this method cannot clean mercaptans over 50-60%, furthermore, these processes require a great number of reagents, solutions and others. Moreover, there are a number of technological problems. In recent years, process of demercaptanization involving catalysts has drawn attention. Herein, aluminum-silicate catalysts are being generally used and the process is conducted at very high temperatures of 800–1000°C involving hydrogen. The most commonly used catalyst is the catalyst system in the form of Co, Mo, Ni–Mo, MoS_2 deposited on aluminum oxide. These catalytic systems showing high activity at high temperatures require large energy costs, their regeneration after catalyst deactivation passes with difficulty. From this standpoint, we set the task of creation of catalytic systems on the basis of local raw materials for processes of a demercaptanization.

2. Methods and Apparatus

For demercaptanization of oil products were obtained catalyst systems based on Mo, Co, V, Ni, P oxides and organic ligands of Co, Mn, Fe deposited Al_2O_3, SiO_2, mordenite, and their activities were revealed in demercaptanization process [12,13]. The obtained results are presented in Table 1. İt should be noted that mercaptan compounds of C_1–C_4 present in the composition of oil products have generally been subject to demercaptanization. Thiophene units of oil were subjected to demercaptanization. Kinetic regularities of the process were studied within a wide range of alterations to key parameters. Initially, the model system was prepared consisting of ethyl mercaptan dissolved in light oil. Some samples of 0.2–0.6 g/l from quantity of an ethyl mercaptan close to real fraction of benzine were prepared. Note that both originally taken substances and obtained compounds were controlled by chromatographic, IR analysis and other methods of analysis. Thus it is possible to identify process indicators, by definition of amount of the obtained free sulfur and saturated hydrocarbons as the main indicators.

Table 1. Study of the activity of the catalyst system in the process of the demercaptanization of oil products.

Catalyst systems	Purification efficiency of benzine	Catalyst systems	Purification efficiency of benzine
MoO_3/Al_2O_3	68	Fe–Mo/Al_2O_3	75
V_2O_5/Al_2O_3	60	Ni–MoO_3/Al_2O_3	70
MoO_3/SiO_2	64	MoO_3/klinoptilolite	75
V_2O_5/SiO_2	60	Co_2O_3/klinoptilolite	72
V–P–O/Al_2O_3	62	Co–Fe/klinoptilolite	92
V–P–O/SiO_2	60	Mn–Fe/klinoptilolite	86
V–Mo–O/Al_2O_3	72	MoO_3/ mordenite	60
V–Mo–O/SiO_2	70	Co_2O_3/ mordenite	60
Co–Mo/Al_2O_3	74	Co–Fe/ mordenite	74
Mn–Fe/Al_2O_3	76	Mn–Fe/ mordenite	78

3. Experimental

The influence of the temperature on conversion level of an ethyl mercaptan, i.e. influence on the degree of purification of benzene has been studied in order to study the kinetic regularities of the process of a demercaptanization. Taking into account the change of ethyl and propyl mercaptans in the low-boiling fraction of 303–3530K of

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benzine in the range 0.2–0.6 g/l influence of temperature on process of demercaptanization was studied.

4. Results and Discussion

The results (table 2–4, fig. 1) show that in the presence of MoO_3/ Al_2O_3, V_2O_5–Mo–O_3 / Al_2O_3, Co_2O_3/ clinoptilolite, MnO_2/ clinoptilolite, etc. at the temperature of 313K ethyl mercaptan conversion varies in the range 70–80% and the temperature increase from 323K to 333K leads to some increase of ethyl mercaptan conversions up to 80–85%.

Table 2. Demercaptanization of ethyl mercaptan with participation of various catalysts, T = 313K, V = 10h^{-1}

Catalytic system	conversion, %	Amount of S, obtained in the demercaptanization		Amount of obtained alkanes	
		q	mol	q	mol
MoO_3/Al_2O_3	72	0.071	0.0022	0.063	0.0011
MoO_3/SiO_2	70	0.070	0.021	0.058	0.0110
V_2O_5–MoO_3/Al_2O_3	75	0.077	0.024	0.069	0.0012
Co–MoO_3 /Al_2O_3	76	0.078	0.025	0.070	0.00121
Fe–MoO_3/Al_2O_3	73	0.074	0.0023	0.062	0.0012
MoO_3/ clinoptilolite	77	0.083	0.0026	0.072	0.0013
Co_2O_3/ clinoptilolite	75	0.077	0.0024	0.077	0.00123
MnO_2/ clinoptilolite	78	0.080	0.025	0.074	0.00125
Co:Fe/ clinoptilolite	96	0.098	0.0030	0.086	0.00154
Mn–Fe/ clinoptilolite	95	0.090	0.028	0.073	0.00146

Table 3. Demercaptanization of ethyl mercaptan with participation of various catalysts, T = 313K, V = 10h^{-1}

Catalytic system	conversion, %	Amount of S, obtained in the demercaptanization		Amount of obtained alkanes	
		q	mol	q	mol
MoO_3/Al_2O_3	76	0.0025	0.0078	0.0012	0.070
MoO_3/SiO_2	74	0.0023	0.074	0.0011	0.062
V_2O_5–MoO_3/Al_2O_3	80	0.0026	0.082	0.0013	0.075
Co–MoO_3 /Al_2O_3	80	0.0026	0.082	0.0013	0.075
Fe–MoO_3/Al_2O_3	73	0.025	0.0078	0.0012	0.070
MoO_3/ clinoptilolite	80	0.026	0.0082	0.0013	0.075
Co_2O_3/ clinoptilolite	78	0.026	0.0081	0.0030	0.072
MnO_2/ clinoptilolite	82	0.027	0.0082	0.0014	0.073
Co:Fe/ clinoptilolite	98	0.016	0.0097	0.0152	0.092
Mn–Fe/ clinoptilolite	97	0.014	0.0096	0.0150	0.090

Only a catalytic system obtained by depositing organic ligand of Mn–Fe and Co–Fe on clinoptilolite at low temperatures allow to reach conversion of ethyl mercaptan about 94–95% and at relatively high temperatures 333°K conversion of 99%. It was studied the influence of the contact time on the activity of those catalytic systems that have a relatively high activity among obtained. As seen from the results of studies (Figure 2), the catalyst system in a contact time of 8–10 minutes shows high activity and provides conversion of ethyl mercaptan to the level of 98–99%. Relatively high demercaptanization of prepared model systems in the table 1 is explained due to the presence in the benzine composition of other impurities.

Table 4. Demercaptanization of ethyl mercaptan with participation of various catalysts, T = 353K, V = 10h^{-1}

Catalytic system	conversion, %	Amount of S, obtained in the demercaptanization		Amount of obtained alkanes	
		q	mol	q	mol
MoO_3/Al_2O_3	80	0.0025	0.082	0.0013	0.075
MoO_3/SiO_2	76	0.0023	0.078	0.0012	0.070
V_2O_5–MoO_3/Al_2O_3	84	0.0026	0.097	0.0013	0.078
Co–MoO_3 /Al_2O_3	85	0.0027	0.098	0.0013	0.079
Fe–MoO_3/Al_2O_3	78	0.0025	0.090	0.0012	0.072
MoO_3/ clinoptilolite	85	0.0027	0.098	0.0013	0.079
Co_2O_3/ clinoptilolite	80	0.0025	0.082	0.0013	0.075
MnO_2/ clinoptilolite	90	0.0026	0.099	0.0013	0.078
Co:Fe/ clinoptilolite	99	0.0031	0.102	0.0016	0.093
Mn–Fe/ clinoptilolite	99	0.102	0.0031	0.0016	0.093

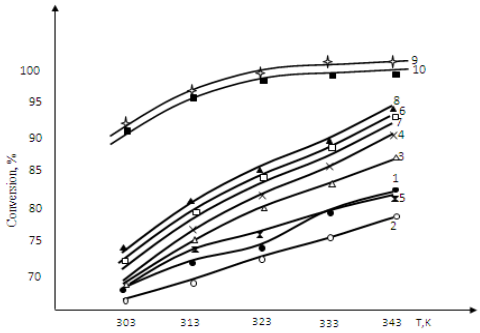

Fig 1. Effect of temperature on the activity of the catalyst system in the process of demercaptanization of ethyl mercaptan. 1–MoO_3/Al_2O_3, 2 – MoO_3/SiO_2, 3 – V_2O_5–MoO_3/Al_2O_3, 4–Co-MoO_3/Al_2O_3, 5–Fe–MoO_3/Al_2O_3, 6–MoO_3/klinop, 7–Co_2O_3/klinop., 8–MnO_2/clinoptilolite, 9 – Co: Fe clinoptilolite, 10 – Mn–Fe/clinoptilolite.

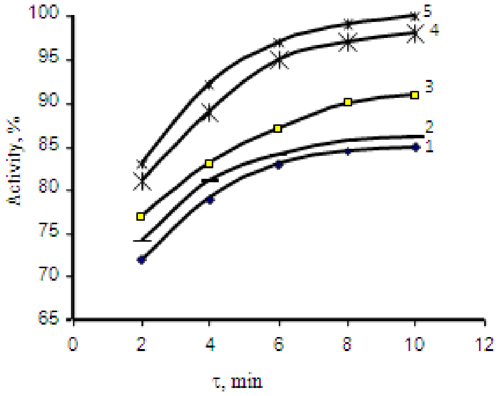

Fig 2. Influence of contact time on the activity of a catalyst in demercaptanization ethyl mercaptan. 1–MoO_3/Al_2O_3, 2–Co–MoO_3/Al_2O_3, 3–MnO_2/clinoptilolite, 4–Mn–Fe/ clinoptilolite, 5–Fe–Co/clinoptilolite.

$$1.\ \underset{A}{C_2H_5SH} + Z \xleftrightarrow[k_{-1}]{k_1} \underset{AZ}{[C_2H_5SH]Z}$$

$$2.\ 2\underset{AZ}{[C_2H_5SH]Z} + O_2 \xleftrightarrow[k_{-2}]{k_2} \underset{R}{ZC_2H_5 \cdot C_2H_5} + S + [H_2O]Z$$

$$3.\ \underset{ZR}{Z\,C_2H_5C_2H_5} \xleftrightarrow[k_{-3}]{k_3} \underset{R}{C_4H_{10}} + Z$$

$$4.\ \underset{AZ}{[\,C_2H_5SH]Z} + O_2 \xleftrightarrow{k_4} 2CO_2 + \underset{M}{S} + 3[H_2O]Z$$

$$5.[H_2O]Z \leftrightarrow H_2O + Z$$

$$6.\ 2\underset{AZ}{[\,C_2H_5SH]Z} + H_2O \xrightarrow[k_6]{} Z \cdot \overset{F}{(C_2H_6)} + \overset{M}{S} + \overset{E}{[C_2H_5OH]Z}$$

$$7.\ 2\underset{AZ}{[\,C_2H_5SH]Z} + O_2 \xrightarrow{k_7} \underset{D}{[CH_2 = CH_2]Z} + [C_2H_5OH]Z$$

$$8.\ [CH_2 = CH_2]Z \xleftrightarrow[k_{-8}]{k_8} D + Z$$

$$9.\ 2\overset{E}{[\,C_2H_5OH]Z} + H_2O \xrightarrow[k_{-9}]{k_9} Z\overset{N}{[C_2H_5OC_2H_5]} + H_2O$$

$$10.\ Z\overset{F}{[C_2H_6]} \xleftrightarrow[k_{-10}]{k_{10}} F + Z$$

$$11.\ Z\overset{N}{(C_2H_5OC_2H_5)} \leftrightarrow \underset{N}{C_2H_5OC_2H_5} + Z$$

$$12.\ Z(C_2H_5OH) \leftrightarrow \underset{E}{C_2H_5OH} + Z$$

$$W_1 = K_1P_A \cdot C_Z - K_{-1}C_{AZ}$$

$$W_2 = K_2P_{O_2} \cdot C_{AZ}^2$$

$$W_3 = K_3C_{ZR} - K_{-3}P_RC_Z$$

$$W_4 = K_4C_{AR}P_{O_2}$$

$$W_5 = K_5C_{Z[H_2o]} - K_{-5}P_{H_2O}C_Z$$

$$W_6 = K_6C_{[AZ]} \cdot P_{H_2O}$$

$$W_7 = K_7C_{AZ}^2 \cdot P_{O_2}$$

$$W_8 = K_8C_{DZ} - K_{-8}P_DC_Z$$

$$W_9 = K_9C_{EZ} - K_{-9}P_EC_Z$$

$$W_{10} = K_{10}C_{FZ} - K_{-10}P_FC_Z$$

$$W_{11} = K_{11}C_{ZN} - K_{-11}P_FC_Z$$

$$W_{12} = K_{12}C_{ZN} - K_{-12}P_NC_Z$$

$$d\frac{AZ}{d\tau} = -K_1P_AC_Z + K_{-1}C_{AZ} - K_2C_{AZ}^2 - K_4C_{AZ} - K_5C_{AZ}^2P_{H_2O} - K_7C_{AZ}^2P_{O_2}$$

$$C_{AZ}^2[K_2 + K_6P_{H_2O} + K_7P_{O_2}] + [K_4 + K_{-1}]C_{AZ} = 0$$

$$C_{AZ} = \frac{\pm[K_{-1} + K_4] \pm \sqrt{(K_2 + K_6P_{H_2O} + K_7P_{O_2})^2 + 4(K_2 + K_6P_{H_2O} + K_7}}{2[K_2 + K_6P_{H_2O} + K_7P_{O_2}]}$$

$$\frac{d_{RZ}}{d\tau} = K_2C_{AZ}^2P_{O_2} + K_{-3}C_ZP_R - K_3C_{RZ}$$

$$C_{RZ} = \frac{K_2C_{AZ}^2P_{O_2} + K_{-3}C_ZP_R}{k_3}$$

$$\frac{dC_{AZ}}{d\tau} = K_7C_{AZ}^2P_{O_2} + K_{-8}C_DC_Z - K_8C_{DZ}$$

$$C_{DZ} = \frac{K_7C_{AZ}^2P_{O_2} + K_{-8}P_DC_Z}{k_8}$$

$$d\frac{C_{EZ}}{d\tau} = K_7C_{EZ} - K_9C_{EZ}^2 + K_9C_{NZ}P_{H_2O} - K_9C_{EZ}^2 - K_7C_{EZ} + K_{-9}C_{NZ}P_{H_2O}$$

$$C_{EZ}^2 = \frac{K_7C_{EZ} + K_9C_{NZ}P_{H_2O}}{k_9}$$

$$d\frac{C_{FZ}}{d\tau} = K_6C_{AZ}^2P_{H_2O} + K_{-10}P_FC_Z - K_{10}C_{FZ}$$

$$C_{FZ} = \frac{K_6C_{AZ}^2P_{H_2O} + K_{-10}P_FC_Z}{k_{10}}$$

$$d\frac{C_{NZ}}{d\tau} = K_9C_{AZ}^2 - K_{-9}C_{NZ}P_{H_2O} + K_{-11}P_NC_Z - K_{11}C_{NZ}$$

$$C_{NZ} = \frac{K_9C_{AZ}^2 + K_{-11}P_NC_Z}{k_{-9} + k_{11}}$$

$$d\frac{C_Z}{d\tau} = K_{-1}C_{AZ} - K_1C_ZP_A + K_{-3}C_{ZR} - K_3P_RC_Z + K_8C_{DZ} -$$
$$- K_{-8}P_DC_Z + K_{10}P_F - K_{-10}C_ZP_F + K_{11}C_{ZN} - K_{-11}C_ZP_N + K_{12}C_{EZ} - K_{-12}P_EC_Z$$

$$C_Z = \frac{K_{-1}C_{AZ} + K_{-3}C_{ZR} + K_8C_{DZ} + K_{10}C_{ZF} + K_{11}C_{ZN} + K_{12}C_{EZ}}{K_1P_A + K_{+}P_R + K_8P_D + K_{-10}P_F + K_{-11}P_N + K_{12}P_E}$$

$$\frac{dC_{[H_2O]t}}{d\tau} = K_2C_{AZ}^2P_{O_2} + K_4C_{AZ}P_{O_2} + K_{-5}P_{H_2O}C_Z - K_5C_{(H_2O)t}$$

$$C_Z + C_{AZ} + C_{RZ} + C_{(H_2O)t} + C_{FZ} + C_{DZ} + C_{EZ} + C_{NZ} = 1$$

$$C_{[H_2O]t} = \frac{K_2P_{AZ}^2P_{O_2} + K_4C_{AZ} + K_{-5}P_{H_2O}C_Z}{K_5}$$

It should be noted that the catalytic systems obtained by precipitation of the anhydrous organic ligands $C_{12}H_{10}Co–FeO_2Cl_2$ and $C_{12}H_{10}MnFeOCl_2$ on the basis of ferrosen of Co–Fe and Mn–Fe on clinoptilolite showed very high activity. At the same time rupture of the stable bond C–S in low-molecular mercaptans occurs due to process of an oxidizing reduction, and as a consequence C–S bond is cut off, resulting in formation of free sulfur and low molecular alkanes.

The mechanism of the demercaptanization process can be represented with the following equations:

A – mercaptans, z–active center of the catalyst, AZ – intermediate complex, R–CH_4H_{10}, F–C_2H_6, D – CH_2 + CH_2, E – C_2H_5OH, N – ether, M – free sulfur.

To estimate the parameters of the obtained kinetic equations can use the Arrhenius equation, i.e., depending of the rate constants and equilibrium constants of the absorption on temperature

$$K_{ef} = K_0^1 \exp(-\frac{E'_{ef}}{RT})$$

$$K_j = K_0^2 \exp(-\frac{Q}{RT})$$

Values of E_{ef} and Q_i – i.e. the activation energy and the absorption heat based on results of experiments were calculated on the computer in Windows XP system by method of optimization of Powell. As optimization criteria is taken the average quadratic changes of experimental and calculated data based on a kinetic model (calculated) (table 5).

$$F=\sum_{i=1}^{n}\sum_{j=1}^{m}\left[\frac{W_i^{\text{тэк}}-W_j^{\text{hes}}}{W_{ij}^{\text{тэк}}}\right]$$

Table 5. Kinetic equation parameters of demercaptanization of ethyl mercaptan.

K	313	323	333	$\ln K_0$	E	Q_i
K_1	$0.625\cdot10^{-3}$	$0.4721\cdot10^{-3}$	$0.3461\cdot10^{-3}$	–5.7	–	36.2
K_{-1}	$0.4421\cdot10^{-3}$	$0.2136\cdot10^{-2}$	$0.0928\cdot10^{-2}$	3.8	–	16.3
K_2	$0.5432\cdot10^{-2}$	$0.8341\cdot10^{-2}$	$1.021\cdot10^{-1}$	–4.6	85.6	–
K_{-2}	$0.3101\cdot10^{-2}$	$0.1036\cdot10^{-2}$	$0.8761\cdot10^{-3}$	6.1	–	20.7
K_3	$0.7141\cdot10^{-3}$	$0.9782\cdot10^{-3}$	$1.3566\cdot10^{-2}$	8.2	–	19.4
K_{-3}	$0.1832\cdot10^{-2}$	$0.4273\cdot10^{-2}$	$0.1836\cdot10^{-2}$	6.4	–	18.1
K_4	$0.4321\cdot10^{-2}$	$0.4321\cdot10^{-2}$	$0.9861\cdot10^{-2}$	–4.2	78.6	–
K_5	$0.3476\cdot10^{-2}$	$0.662\cdot10^{-2}$	$0.8792\cdot10^{-2}$	–4.0	–	17.2
K_{-5}	$0.1832\cdot10^{-2}$	$0.6741\cdot10^{-2}$	$0.0524\cdot10^{-2}$	7.4	–	15.4
K_6	$0.5831\cdot10^{-2}$	$0.8470\cdot10^{-2}$	$1.1231\cdot10^{-1}$	5.2	44.2	–
K_7	$0.6482\cdot10^{-2}$	$0.9631\cdot10^{-2}$	$1.3645\cdot10^{-1}$	7.1	74.3	–
K_8	$0.3241\cdot10^{-3}$	$0.5882\cdot10^{-3}$	$0.8631\cdot10^{-3}$	5.4	–	18.4
K_{-8}	$0.8214\cdot10^{-3}$	$0.6134\cdot10^{-3}$	$0.3243\cdot10^{-3}$	3.7	–	16.6
K_9	$0.7431\cdot10^{-2}$	$0.9361\cdot10^{-2}$	$1.3436\cdot10^{-1}$	5.8	56.2	–
K_{10}	$0.4632\cdot10^{-2}$	$0.8472\cdot10^{-2}$	$1.2637\cdot10^{-1}$	4.7	–	12.4
K_{-10}	$0.6334\cdot10^{-3}$	$0.4641\cdot10^{-3}$	$0.2144\cdot10^{-3}$	6.4	–	15.6
K_{11}	$0.2112\cdot10^{-2}$	$0.3641\cdot10^{-2}$	$0.5634\cdot10^{-2}$	–5.3	–	13.3
K_{-11}	$0.5542\cdot10^{-2}$	$0.4133\cdot10^{-2}$	$0.2234\cdot10^{-2}$	4.6	–	14.8
K_{12}	$0.4356\cdot10^{-2}$	$0.7354\cdot10^{-2}$	$0.9421\cdot10^{-2}$	–4.4	–	15.8
K_{-12}	$0.7648\cdot10^{-2}$	$0.5331\cdot10^{-2}$	$0.3754\cdot10^{-2}$	5.4	–	18.2

5. Conclusions

1.On the basis of the studied kinetic regularities data the kinetic model for the of heterogeneous catalystic process of demercaptanization of oil is developed.

2.The values of the rates of accumulation of reaction products and consumption of the initial hydrocarbon and activation energy of reactions are calculated.

3.A kinetic model is pretty well explains the experimental results and the relative error does not exceed 7–8%.

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