

## Thermal Decomposition of a Mixture of Tar with Primary Coal Tar with the Additives of Iron Compounds

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**Abstract**—Studies on the thermal destruction of a mixture of vacuum residue (tar) and primary coal tar (PCT) with and without  $\text{Fe}_3\text{O}_4$  and  $\beta\text{-FeOOH}$  catalytic additives, which were performed using thermogravimetry and differential scanning calorimetry in an inert atmosphere at a heating rate of 10 K/min, are reported. Based on the results of thermogravimetric analysis and the kinetic parameters of the process, activation energies were calculated, and these activation energies can be used in the development of methods for the technological calculation of reactors and the selection of construction materials for their manufacture.

**Keywords:** primary coal tar, tar, destruction, nanocatalytic additive

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Reliable methods for the technological calculation of reactors, the design of new types of equipment, and the selection of construction materials and data on the rate constants of main reactions, activation energies, and other parameters determining the macrokinetics of a process are necessary for the development of highly efficient processes of the coking, catalytic cracking, and visbreaking of solid and heavy hydrocarbon raw materials. Nonisothermal research methods, in particular, thermogravimetric and differential thermal analysis, are used in order to optimize a process. The use of new inexpensive disposable catalysts in various processes is promising and relevant because these nanocatalysts are consumed in much lower concentrations.

### EXPERIMENTAL

To determine the kinetic parameters of a thermal degradation process, we carried out the thermogravimetric and differential thermal analysis of a mixture of tar (vacuum residue) and primary coal tar (PCT) with and without the iron-based catalytic additives  $\beta\text{-FeOOH}$  and  $\text{Fe}_3\text{O}_4$ .

Tar (vacuum residue) from the Pavlodar refinery, primary coal tar, and catalytic additives based on iron

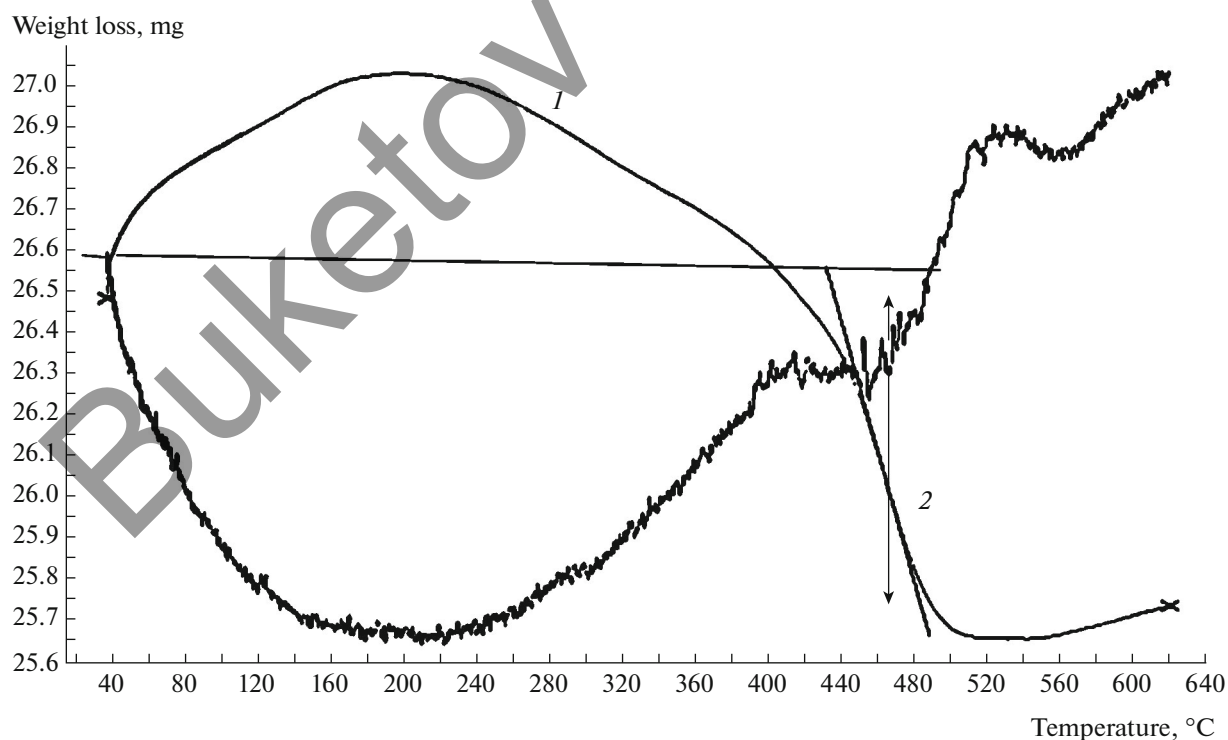
were used in the experiments. The amounts of added PCT and tar were 20 and 80%, respectively. The particle size of dispersed catalytic additives was 20 nm. The use of such a quantity of PCT was explained by the fact that the addition of polyaromatic compounds (anthracene, phenanthrene, etc.) can prevent recombination reactions until the saturation of coal associates with hydrogen [1–3]. In this case, oil and coal radicals were stabilized and atomic hydrogen was generated. The polyaromatic hydrocarbon constituents of PCT play the role of activators and hydrogen carriers under the conditions of the thermochemical processing of tar. In this case, PCT plays the role of an inhibitor additive to tar for preventing secondary condensation reactions of the primary degradation products of the mixture.

Table 1 summarizes the physicochemical properties of the tar (TOO Pavlodar Petrochemical Plant) and the technical characteristics of the primary coal tar (TOO Sary-Arka Spetzkoks) are presented in.

When studying the thermal decomposition process of a mixture of tar with PCT and catalytic additives, we found that it is a set of physicochemical transformations and chemical reactions occurring in certain temperature ranges. These processes cannot be studied using classical methods of chemical kinetics because

**Table 1.** Physicochemical properties of tar and technical properties of PCT

Characteristic	Tar	Characteristic	Primary coal tar
Density at 20°C, kg/m <sup>3</sup>	967.0	Volume fraction of water, %	10.4
Pour point, °C	25	Density at 20°C, kg/m <sup>3</sup>	1042
Viscosity at 70°C, Pa s	1.7	to 180°C	3
Coking capacity, %	11.3	180–230°C	7.2
Concentration, %:		230–270°C	15.1
asphaltene	9.8	270–300°C	17.1
vtar	12.5	–	–
oil	78.2	Final boiling temperature, °C:	
Elemental composition, wt %:		vapor	315
C	84.10	liquid	390
H	10.82	Yield of pitch, %	50
S	3.15	Weight fraction of substances insoluble in toluene, %	3.8
N	0.71	Weight fraction of substances insoluble in quinolone, %	None
O	1.22	Ash content, %	0.1
Boiling away to 520°C, %	12.3	Concentration, %	
Metal content, g/t:		phenol	>20
vanadium	210.0	naphthalene	Traces
nickel	74.0		

**Fig. 1.** Results of studies of the thermal destruction of a mixture of tar in the presence of PCT using DTA: (1) TG and (2) DTG.

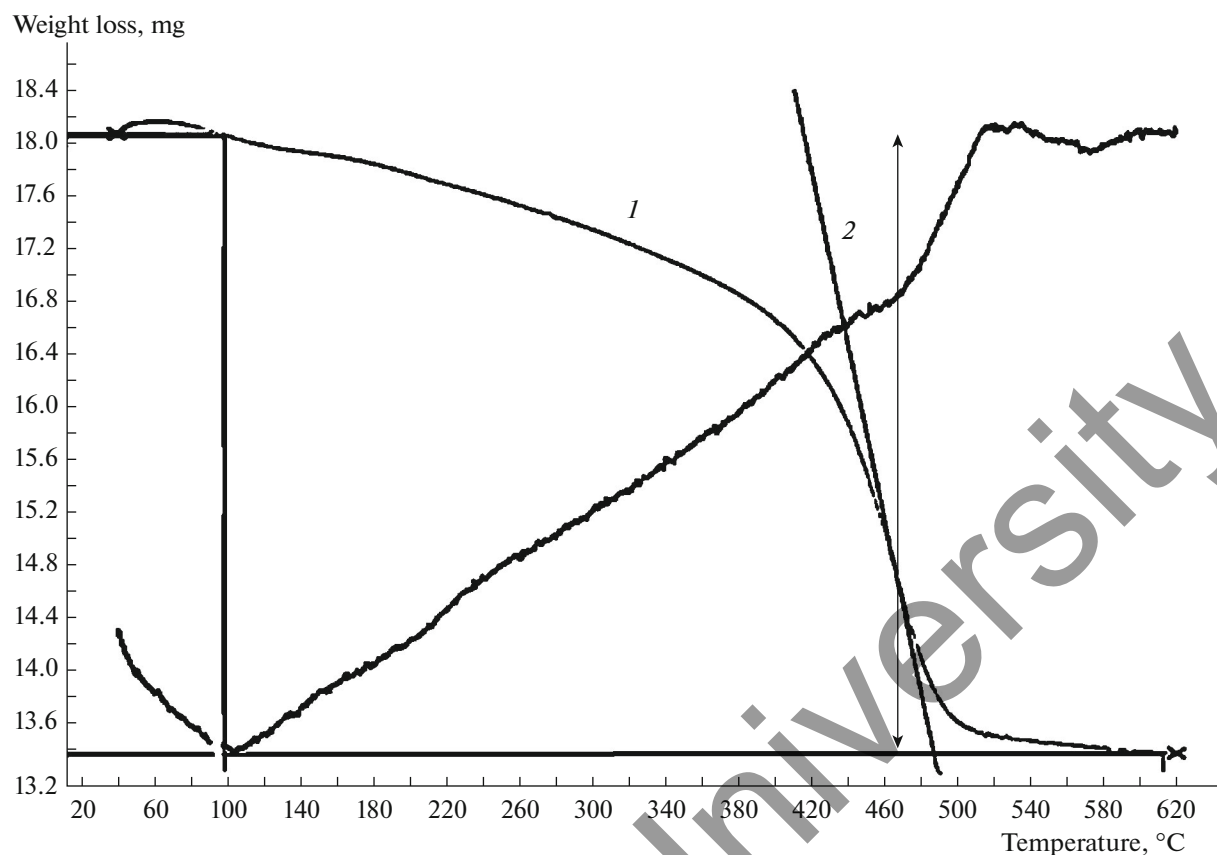


Fig. 2. Results of studies of the thermal destruction of a mixture of tar and PCT in the presence of a catalytic additive  $\beta$ -FeOOH (1%) using DTA: (1) TG and (2) DTG.

Table 2. Results of the processing of thermal destruction curves for a mixture of tar and PCT

Entry	$\tau$ , min	$m_i$ , mg	$m_i - m_{\max}$ , mg	$r_i$ , mg/min	$r_i/m_{i\max} - m_i$ , mg	$-\ln(r_i/m_{i\max} - m_i)$	$t$ , °C	$10^3/T$ , K <sup>-1</sup>
1	14.2	—	—	—	—	—	161	2.30
2	17.3	2.995	1.324	0.467	0.353	1.042	196	2.13
3	20.5	3.015	1.305	0.014	0.011	4.538	230	1.99
4	23.7	3.088	1.232	0.025	0.021	3.881	265	1.86
5	27.0	3.170	1.150	0.028	0.024	3.709	299	1.75
6	30.2	3.253	1.067	0.027	0.026	3.665	334	1.65
7	33.5	3.345	0.975	0.029	0.030	3.499	368	1.56
8	36.8	3.467	0.853	0.051	0.060	2.808	403	1.48
9	40.1	3.675	0.644	0.010	0.016	4.144	437	1.41
10	43.4	4.142	0.178	0.099	0.558	0.584	472	1.34
11	46.7	4.320	—	—	—	—	506	1.28

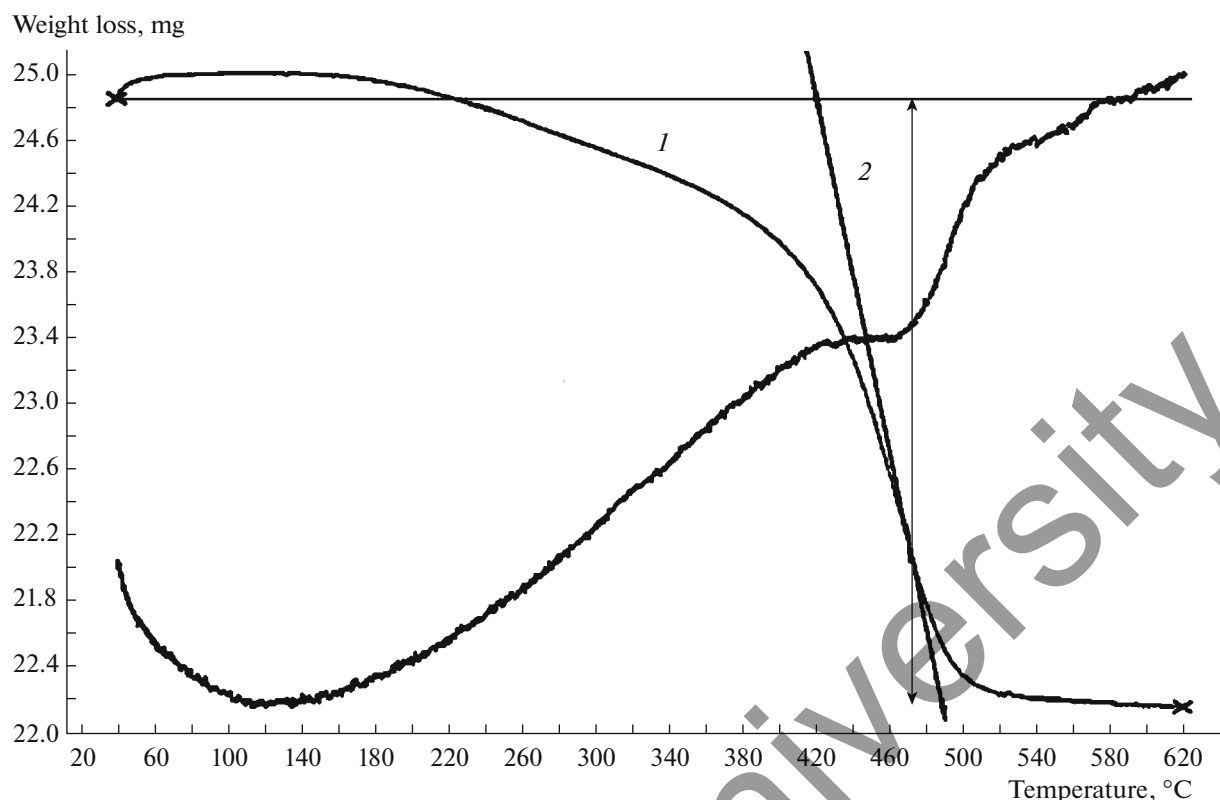


Fig. 3. Results of studies of the thermal destruction of a mixture of tar and PCT in the presence of a catalytic additive  $\text{Fe}_3\text{O}_4$  (1%) using DTA: (1) TG and (2) DTG.

of a large number of simultaneously occurring consecutive and parallel reactions with the participation of a variety of unidentified substances [4–6].

Thermogravimetric analysis in order to determine the effect of catalytic additives on the kinetic parameters of the process of thermal destruction of a mixture of tar and PCT in the presence of catalytic additives and without them were performed using a Labsys Evo Setaram instrument (France) [7]. The instrument consisted of TG thermogravimetric balance connected to ATD and DSC converters, metal-resistance furnace, and multitask software to control various units [8, 9].

Two identical crucibles of  $\text{Al}_2\text{O}_3$  with a volume of 100  $\mu\text{l}$  were used for performing thermal analysis; the construction of the crucibles made it possible to perform temperature measurements directly in a test sample. A weighed preliminarily prepared sample (tar and PCT without a catalyst or in the presence of 0.5, 1, or 1.5% catalytic additive) was loaded in the first crucible, and the second crucible was empty as a reference. The TG/DSC measurements were performed at a constant heating rate of 10 K/min with monitoring weight changes. The prepared crucibles were placed in an electric furnace and heated from 32 to 458 °C with

a constant heating rate of 10 K/min, and weight changes were measured. Nitrogen was used as an inert atmosphere.

The processes were carried out under comparable conditions because the weight of the sample and the particle size distribution of the substance affect the results of the analysis. The shape of thermal analysis

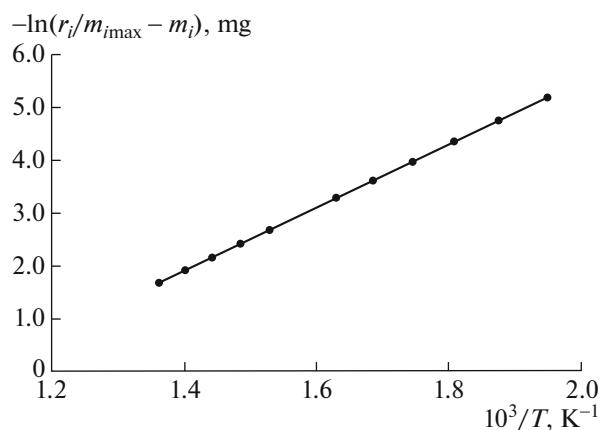


Fig. 4. Dependence of the rate constant on the reciprocal of temperature for a mixture of tar with PCT.

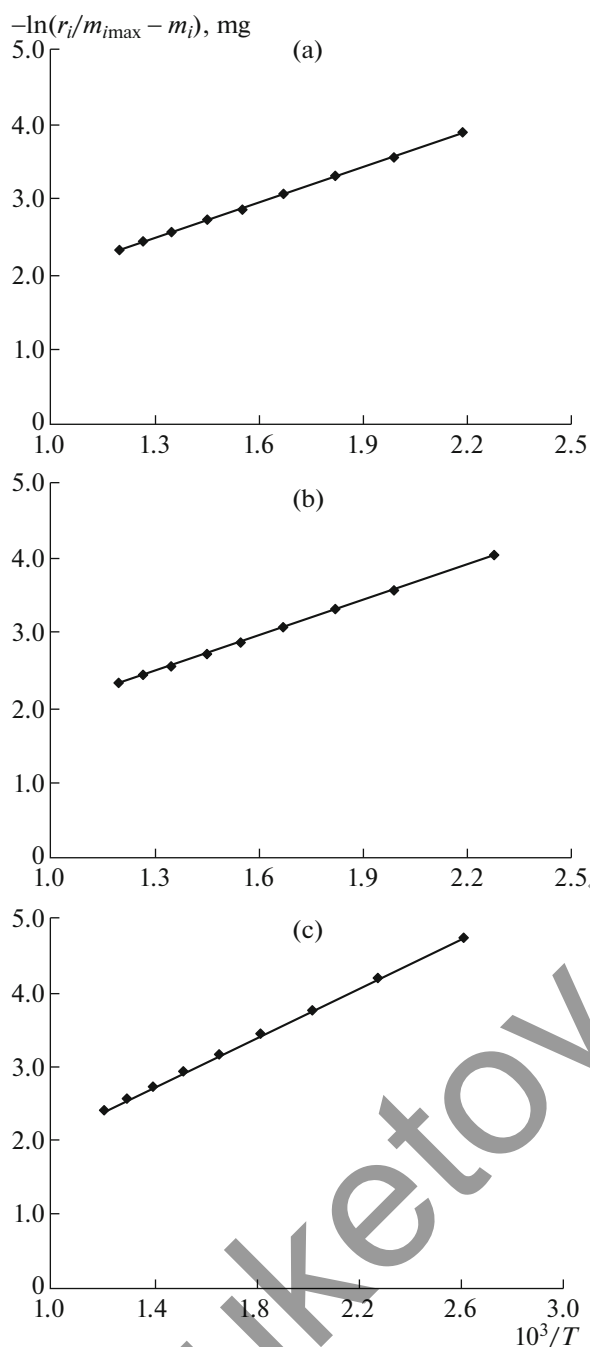


Fig. 5. Dependences of the rate constant on the reciprocal of temperature for the mixtures of (a) tar + PCT +  $\beta$ -FeOOH (0.5%), (b) tar + PCT +  $\beta$ -FeOOH (1%), and (c) tar + PCT +  $\beta$ -FeOOH (1.5%).

curves depends on gas evolution conditions: if the rate of formation of gaseous substances was higher than the rate of their removal, the gaseous products accumulated inside the sample to affect the course of thermal destruction. To avoid the accumulation of gases in the derivatograph, their suction was provided.

## RESULTS AND DISCUSSION

As a result of the thermogravimetric study, TG, DTG, and DTA curves were obtained, which showed a decrease in the rate of weight loss of the samples with temperature and the thermal effects of the processes. Figures 1–3 show the thermogravimetric curves of thermal destruction of a mixture of tar and PCT without catalysts and in the presence of the catalytic additives (C, 1%) of  $\beta$ -FeOOH and  $\text{Fe}_3\text{O}_4$ .

The temperatures at which changes in the rates of weight loss occurred and their maximum values in the DTG curves were determined in a study of the dynamics of thermal destruction processes of a mixture of tar and PCT without catalysts and in the presence of the catalytic additives  $\beta$ -FeOOH and  $\text{Fe}_3\text{O}_4$ .

Table 2 summarizes the results of the processing of thermal destruction curves of a mixture of tar and PCT.

The parameters  $a_i = \ln k_{0i}$  and  $b_i = E_i/R$  were chosen by linearizing the equation. Based on the data of Table 2, the dependence of  $-\ln[r_i/(m_{i_{\max}} - m_i)]$  on  $10^3/T$  was plotted for the sample shown in Fig. 4, and the curves obtained were processed by a least squares technique. The processing of curves shown in Figs. 5a–5c using a least squares technique showed the following results for tar + PCT, %: stage I,  $\ln k_0 = -4.2574$ ;  $k = -4.2574 \times 10^{-3} \text{ min}^{-1}$ ; and  $E_1 = 38.5595 \text{ kJ/mol}$  (the process occurs in the diffusion region) and stage II,  $\ln k_0 = -5.4591$ ;  $k = -5.4591 \times 10^{-3} \text{ min}^{-1}$ ; and  $E_2 = 110.16 \text{ kJ/mol}$ .

Tables 3–5 summarize the results of the processing of thermal destruction curves for a mixture of tar and PCT in the presence of the catalytic additive  $\beta$ -FeOOH.

The parameters  $a_i = \ln k_{0i}$  and  $b_i = E_i/R$  were selected by linearizing a rate equation [5]. Based on the data given in Tables 3–5, the dependence of  $\ln[r_i/(m_{i_{\max}} - m_i)]$  on  $10^3/T$  was plotted for each sample (Figs. 5a–5c), and the curves were processed using a least squares technique. Tables 6–8 summarize the results of the processing of thermal decomposition curves for a mixture of tar and PCT in the presence of the catalytic additive  $\text{Fe}_3\text{O}_4$ .

The parameters  $a_i = \ln k_{0i}$  and  $b_i = E_i/R$  were selected by linearizing a rate equation based on the data given in Tables 6–8; the dependence of  $-\ln[r_i/(m_{i_{\max}} - m_i)]$  on  $10^3/T$  was plotted for each particular sample (Fig. 6a–6c), and the curves were processed using a least squares technique.

Table 9 shows comparative data to indicate that the activation energy increased from 78.610 to 93.201 kJ/mol as the concentration of the catalytic additive  $\beta$ -FeOOH was increased from 0.5 to 1%, and the activation energy decreased from 93.201 to 65.641 kJ/mol as the concentration of the catalyst FeOOH was increased from 1 to 1.5%.

Similar data were obtained for the catalyst  $\text{Fe}_3\text{O}_4$ : as the  $\text{Fe}_3\text{O}_4$  catalyst content was increased from 0.5 to

**Table 3.** Results of the processing of thermal destruction curves for a mixture of tar, PCT, and  $\beta$ -FeOOH (0.5%)

Entry	$\tau$ , min	$m_i$ , mg	$m_i - m_{\max}$ , mg	$r_i$ , mg/min	$r_i/m_{\max} - m_i$ , mg	$-\ln(r_i/m_{\max} - m_i)$	$t$ , °C	$10^3/T$ , K <sup>-1</sup>
1	11.8	—	—	—	—	—	135	2.45
2	16.1	0.056	3.552	0.019	0.005	5.229	183	2.19
3	20.5	0.180	3.428	0.031	0.009	4.699	230	1.99
4	25.0	0.328	3.280	0.038	0.012	4.457	277	1.82
5	29.4	0.500	3.108	0.049	0.016	4.140	324	1.67
6	33.9	0.778	2.830	0.106	0.038	3.283	371	1.55
7	38.5	1.472	2.136	0.247	0.115	2.159	419	1.45
8	43.0	3.024	0.584	0.228	0.391	0.940	466	1.35
9	47.7	3.523	0.085	0.059	0.699	0.358	513	1.27
10	52.3	3.577	0.031	0.011	0.335	1.094	560	1.20
11	56.9	3.608	—	—	—	—	607	1.14

**Table 4.** Results of the processing of thermal destruction curves for a mixture of tar, PCT, and  $\beta$ -FeOOH (1%)

Entry	$\tau$ , min	$m_i$ , mg	$m_i - m_{\max}$ , mg	$r_i$ , mg/min	$r_i/m_{\max} - m_i$ , mg	$-\ln(r_i/m_{\max} - m_i)$	$t$ , °C	$10^3/T$ , K <sup>-1</sup>
1	4.2	—	—	—	—	—	55	3.05
2	9.5	0.184	0.184	4.828	0.029	0.006	110	2.61
3	14.6	0.316	0.316	4.697	0.034	0.007	165	2.28
4	19.7	0.532	0.532	4.480	0.044	0.010	220	2.02
5	24.8	0.781	0.781	4.232	0.052	0.012	275	1.82
6	30.0	1.087	1.087	3.925	0.068	0.017	331	1.66
7	35.3	1.519	1.519	3.493	0.164	0.047	386	1.52
8	40.6	2.828	2.828	2.185	0.310	0.142	441	1.40
9	45.9	4.808	4.808	0.204	0.201	0.982	496	1.30
10	51.3	4.936	4.936	0.077	0.016	0.214	551	1.21
11	56.8	5.013	5.013	—	—	—	606	1.14

**Table 5.** Results of the processing of thermal destruction curves for a mixture of tar, PCT, and  $\beta$ -FeOOH (1.5%)

Entry	$\tau$ , min	$m_i$ , mg	$m_i - m_{\max}$ , mg	$r_i$ , mg/min	$r_i/m_{\max} - m_i$ , mg	$-\ln(r_i/m_{\max} - m_i)$	$t$ , °C	$10^3/T$ , K <sup>-1</sup>
1	12.0	—	—	—	—	—	138	2.43
2	16.3	0.037	4.780	0.020	0.004	5.501	185	2.18
3	20.6	0.176	4.641	0.040	0.009	4.762	232	1.98
4	25.0	0.372	4.445	0.051	0.012	4.465	278	1.81
5	29.4	0.612	4.205	0.076	0.018	4.014	325	1.67
6	33.9	1.063	3.754	0.175	0.046	3.069	372	1.55
7	38.4	2.165	2.652	0.339	0.128	2.057	419	1.45
8	42.9	4.105	0.712	0.276	0.388	0.947	466	1.35
9	47.5	4.649	0.168	0.071	0.425	0.856	512	1.27
10	52.0	4.747	0.070	0.018	0.255	1.367	559	1.20
11	56.6	4.817	—	—	—	—	606	1.14

**Table 6.** Results of the processing of thermal destruction curves for a mixture of tar, PCT, and Fe<sub>3</sub>O<sub>4</sub> (0.5%)

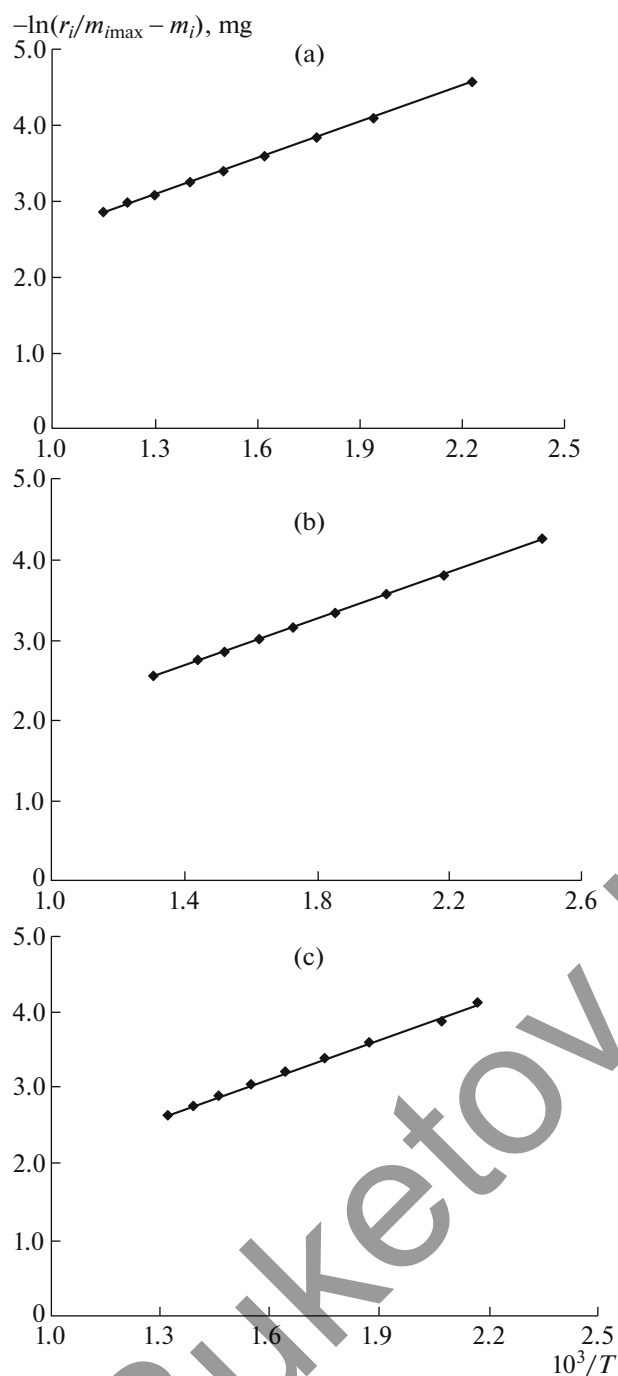
Entry	$\tau$ , min	$m_i$ , mg	$m_i - m_{\max}$ , mg	$r_i$ , mg/min	$r_i/m_{i\max} - m_i$ , mg	$-\ln(r_i/m_{i\max} - m_i)$	$t$ , °C	$10^3/T$ , K <sup>-1</sup>
1	12.6	—	—	—	—	—	144	2.40
2	15.9	0.009	3.794	0.088	0.023	3.762	180	2.21
3	19.2	0.058	3.745	0.024	0.006	5.051	216	2.05
4	22.6	0.168	3.635	0.030	0.008	4.807	252	1.91
5	25.9	0.263	3.540	0.030	0.008	4.779	288	1.78
6	29.4	0.386	3.417	0.037	0.011	4.528	324	1.68
7	32.8	0.524	3.279	0.069	0.021	3.860	359	1.58
8	36.2	0.849	2.954	0.150	0.051	2.983	395	1.50
9	39.7	1.583	2.220	0.330	0.149	1.906	431	1.42
10	43.2	3.152	0.651	0.317	0.487	0.719	467	1.35
11	46.6	3.803	—	—	—	—	503	1.29

**Table 7.** Results of the processing of thermal destruction curves for a mixture of tar, PCT, and Fe<sub>3</sub>O<sub>4</sub> (1%)

Entry	$\tau$ , min	$m_i$ , mg	$m_i - m_{\max}$ , mg	$r_i$ , mg/min	$r_i/m_{i\max} - m_i$ , mg	$-\ln(r_i/m_{i\max} - m_i)$	$t$ , °C	$10^3/T$ , K <sup>-1</sup>
1	12.0	—	—	—	—	—	138	2.43
2	15.4	0.047	3.709	0.022	0.006	5.105	175	2.23
3	18.9	0.145	3.612	0.032	0.009	4.729	213	2.06
4	22.4	0.281	3.475	0.041	0.012	4.448	250	1.91
5	25.9	0.432	3.325	0.041	0.012	4.396	287	1.79
6	29.4	0.586	3.170	0.049	0.015	4.175	325	1.67
7	33.0	0.788	2.968	0.076	0.026	3.659	362	1.58
8	36.6	1.132	2.624	0.162	0.062	2.782	399	1.49
9	40.2	1.945	1.811	0.306	0.169	1.779	436	1.41
10	43.8	3.356	0.400	0.249	0.623	0.473	474	1.34
11	47.4	3.756	—	—	—	—	511	1.28

**Table 8.** Results of the processing of thermal destruction curves for a mixture of tar, PCT, and Fe<sub>3</sub>O<sub>4</sub> (1.5%)

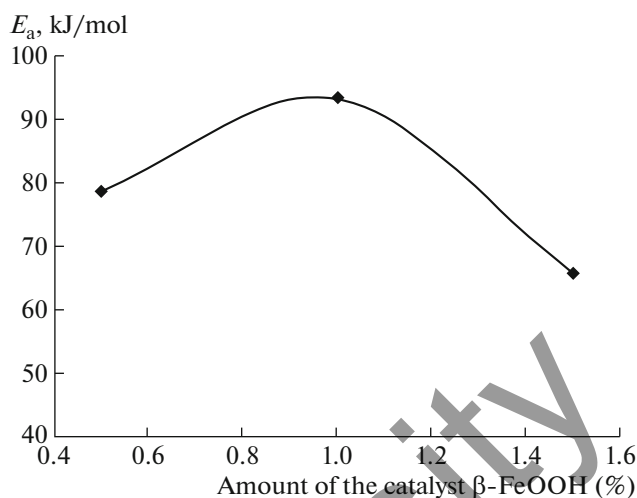
Entry	$\tau$ , min	$m_i$ , mg	$m_i - m_{\max}$ , mg	$r_i$ , mg/min	$r_i/m_{i\max} - m_i$ , mg	$-\ln(r_i/m_{i\max} - m_i)$	$t$ , °C	$10^3/T$ , K <sup>-1</sup>
1	13.1	—	—	—	—	—	150	2.36
2	16.6	0.031	3.882	0.017	0.004	5.421	187	2.17
3	20.0	0.123	3.789	0.033	0.009	4.728	224	2.01
4	23.5	0.255	3.657	0.042	0.012	4.457	262	1.87
5	27.0	0.405	3.507	0.047	0.013	4.310	299	1.75
6	30.5	0.587	3.325	0.064	0.019	3.951	336	1.64
7	34.1	0.848	3.064	0.103	0.034	3.389	373	1.55
8	37.7	1.306	2.606	0.226	0.087	2.444	410	1.46
9	41.3	2.453	1.459	0.337	0.231	1.467	448	1.39
10	44.9	3.760	0.152	0.197	1.292	0.256	485	1.32
11	48.5	3.912	—	—	—	—	522	1.26



**Fig. 6.** Dependences of the rate constant on the reciprocal of temperature for the mixtures of (a) tar + PCT +  $\text{Fe}_3\text{O}_4$  (0.5%), (b) tar + PCT +  $\text{Fe}_3\text{O}_4$  (1%), and (c) tar + PCT +  $\text{Fe}_3\text{O}_4$  (1.5%).

1%, the activation energy decreased from 95.941 to 93.281 kJ/mol, and the activation energy increased from 93.281 to 109.301 kJ/mol with increasing the  $\text{Fe}_3\text{O}_4$  catalytic additive concentration from 1.0 to 1.5% (Fig. 4).

According to the data given in Table 8, the dependences of the activation energy on the catalyst concen-



**Fig. 7.** Dependence of activation energy ( $E_a$ , kJ/mol) on the amount of the catalytic additive  $\beta\text{-FeOOH}$  (%).

tration were plotted (Figs. 7, 8); from these graphs, it follows that the lowest activation energy was observed with the addition of 1% nanocatalysts.

This fact suggests that the use of new relatively cheap disposable catalysts, which were not studied previously in the processes of thermal destruction, is more effective because the activation energy was significantly decreased with the use of a 1% concentration instead of commonly used 5%.

The results of the thermogravimetric analysis can be used to study the process of hydrogenation, which requires the development of reliable methods for the technological calculation of a reactor based on the known values of rate constants for the main reactions, activation energies, and other parameters determining the macrokinetics of a thermal destruction process. So-called kinetic models with distributed parameters can be used for the analysis of complex reactions. For example, a kinetic model with distributed activation energy [2] adequately reflects the thermolysis of coal and oil residues with the formation of volatile substances. Thermal destruction of heavy petroleum residues with the formation of volatile products results from the occurrence of an indefinite number of parallel first-order reactions. Therefore, the task of kinetic

**Table 9.** Calculated rate constants and activation energies of the thermal destruction of a mixture of tar and PCT without catalysts and in the presence of the catalytic additives  $\beta\text{-FeOOH}$  and  $\text{Fe}_3\text{O}_4$

Entry	$\beta\text{-FeOOH}$ , %	$E_1$ , kJ/mol	$\text{Fe}_3\text{O}_4$ , %	$E_2$ , kJ/mol
1	—	110.161	—	110.161
2	0.5	78.610	0.5	95.943
3	1.0	93.201	1.0	93.281
4	1.5	65.640	1.5	109.301

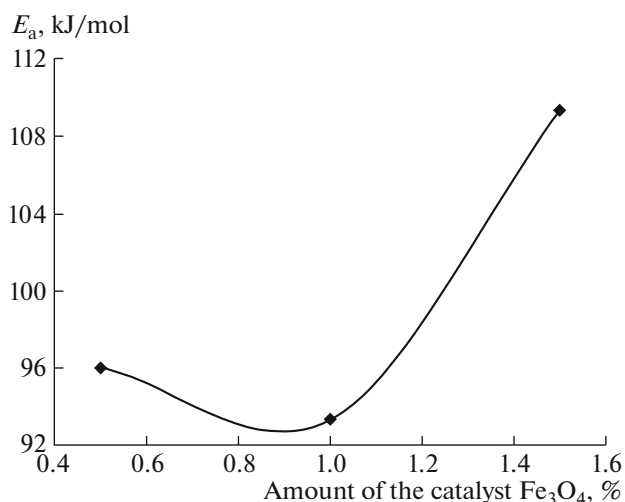


Fig. 8. Dependence of activation energy ( $E_a$ , kJ/mol) on the amount of the catalytic additive  $\text{Fe}_3\text{O}_4$  (%).

analysis was to estimate the parameters  $k_0$  and activation energy ( $E$ ) for the thermal cracking of oil residues from various fields. The results presented in this article are consistent with published data [2, 6].

### CONCLUSIONS

Thus, according to thermogravimetric analysis data, we calculated the kinetic parameters of thermal destruction processes in a mixture of tar and PCT without catalysts and in the presence of a catalytic additive in the form of  $\text{Fe}_3\text{O}_4$  and  $\beta\text{-FeOOH}$  powders (in amounts of 0.1; 0.5; and 1%) in a temperature range of 32–458°C. We found that the processes of thermal destruction of a mixture of tar and PCT without catalysts and in the presence of catalytic additives in the above temperature range occurred in two stages. The rate constants and activation energies of thermal destruction processes were determined.

Similarly, for the catalytic additive  $\text{Fe}_3\text{O}_4$ , the activation energy decreased from 95.941 to 93.281 kJ/mol

as the  $\text{Fe}_3\text{O}_4$  content was increased from 0.5 to 1%, and the activation energy increased from 93.281 to 109.301 kJ/mol with an increase in the concentration of the catalytic additive  $\text{Fe}_3\text{O}_4$  from 1 to 1.5%.

We demonstrated that thermogravimetric analysis allows one to study successfully the influence of various factors on the thermochemical processing of tar in the presence of catalytic additives.

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