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Dearomatization of the Kerosene Fraction: Kinetic Studies

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Abstract

The kinetics of dearomatization of a kerosene fraction processed using a zeolite catalyst (0.9 HZSM-5) at different temperatures (160-200°C), the molar ratio between the aromatic hydrocarbons present in the kerosene fraction and *n*-decene (1:(0.5-4)), and the reaction time (1-3 h) were studied. Based on the obtained data, a kinetic model for kerosene dearomatization is proposed. It is assumed that the single-center Riedel mechanism is followed. The stage associated with the interaction between *n*-decene adsorbed on the surface of the catalyst containing aromatic compounds and *n*-decene present in the volume is identified as the limiting sage of the dearomatization process.

Keywords

Kerosene fraction; dearomatization; zeolite catalyst; n-decene; kinetic regularities

1. Introduction

The current and future environmental demands limit the arene content in gasoline and kerosene. The expected performance cannot be achieved using traditional hydrotreatment technologies. Hence, special catalytic hydrodearomatization processes are carried out at high pressures. The execution of these special processes requires the use of expensive catalysts. Although the processes of extraction dearomatization proceed at relatively low temperatures and pressure, the extractants used in most cases are harmful to the environment.

The process of dearomatization of gasoline and kerosene fractions was studied following the alkylation and reforming processes. Catalytic cracking of gasoline containing propane-propylene, and butane-butylene fractions was conducted using various zeolite-containing catalysts. This allows helps increase the yield of gasoline. The use of higher α-olefins and styrene makes it possible to obtain components of environmentally friendly fuels and raw materials for pyrolysis, with the production of oligoalkylates, which are the basis for the production of synthetic industrial oils and phenylarylethanes, which are dielectric liquids [1-8].

It has been previously reported [5] that the kerosene fraction contains up to 22.0% (wt.%) of aromatic hydrocarbons (chain length: C6-C10). The kinetics of the process can be influenced by various processes such as re-alkylation and dealkylation.

Dearomatization of oil fractions with higher-order olefins (chain length: C8-C12) and the oligomerization processes of these fractions were studied in the liquid phase in the presence of ZSM-5 zeolite.

The reagents used were pre-dried with freshly baked calcium chloride and distilled.

2. Experiments

We studied the kerosene fraction, and the physical and chemical characteristics of the fraction are presented in Table 1 and Table 2.

Table 1 Basic physical and chemical properties of the kerosene fraction.

Density at 20°C, kg/m^3	Fractional composition, °C				Kinematic viscosity at 20°C, мм^2/с	Acidity, mL KOH/100 ml fuel	Iodine number, g I_2/100 mL fuel	temperature, °C	
	The beginning of the boil.	10% boil-off at	50% boil-off at	90% boil-off at				outbreaks	crystallization
794	144	162	185	224	1.44	0.13	0.8	36	-58

Table 2 Hydrocarbon composition of the kerosene fraction.

Compound	Quantity, mass%
1	2
Paraffins	50.5
Naphthenes	20.0
Olefins	0.65
Aromatic compounds, including:	22.0
Monoalkylbenzenes	4.04
Dialkylbenzenes	5.02
Trialkylbenzenes	12.70
Tetraalkylbenzenes	0.24

Condensed aromatic compounds (naphthalene, methylnaphthalene)	0.85
Resins	6.0

We used the commercial zeolite ZSM-5 with silicate module 90 to conduct our experiments. In the proses of preparation of the H-forms of high-silica zeolites, the sodium forms of the samples were loaded into a reactor equipped with a stirrer. The reactor was filled with a solution of ammonium sulfate. The final suspension was heated in the temperature range of 80-85°C, and it was constantly mixed for 1.5-2 h. During this period, the ion exchange process for sodium in the zeolite was in progress. The zeolite suspension obtained after the ion exchange process was washed using hot water (55°C) in a vacuum filter. Subsequently, it was dehydrated.

The dehydrated zeolite sample was dried in the temperature range of 120-140°C. The dried zeolite was calcined in a muffle furnace in the temperature range of 550-600°C over a period of 3 h. A solution of ammonium sulfate was used for repeated ion exchange for the $NH4^+$ ions. The degree of exchange of Na^+ ions with $NH4^+$ exceeded 90%.

The content of residual sodium in the ion-exchange zeolite was determined using an FPP-1 flame ionization photometer.

Kinetics of the *n*-decene alkylation process was studied using a complete mixing reactor, which is an autoclave with a capacity of 100 mL. The temperature in the reaction zone was maintained to an accuracy of ±2.0°C. The kerosene fraction, *n*-decene, and the catalyst were placed in the reactor. The reaction time was recorded from the moment the agitator was switched on. Sampling for GJ chromatographic analysis was performed at regular intervals using a syringe.

The raw materials and reaction products were analyzed using the gas-liquid chromatography technique using a chromatograph equipped with a thermal conductivity detector (a steel column; length: 3 m; internal diameter: 3 mm). Dodecan was used as the internal standard.

The qualitative and quantitative composition of the kerosene fraction was determined using the Auto System XL chromatograph (column height: 100 m).

3. Results and Discussion

The alkylation of benzene with *n*-decene was studied previously, and the mixing intensity was determined. It was observed that the reaction speed did not depend on the number of revolutions of the agitator. When the speed of rotation of the agitator is 800 rpm, the reaction speed is not influenced by the intensity of mixing (Table 3). This indicates that the process of external diffusion does not influence the process under study.

Table 3 Effect of rotation speed on the initial rate of the alkylation reaction.

Conditions	Stirrer rotation speed, min^{-1}				
	200	400	600	800	1000
$-w$, $mol \cdot l^{-1} \cdot h^{-1} \cdot g\text{-}cat^{-1}$	2.1	6.3	24.3	25.6	25.7

Experimental conditions: Temperature: 160°C, mole ratio benzene:*n*-decene = 6:1, contact time: 1 h.

All kinetic experiments were performed at this mixing intensity.

During the analysis of the kinetic data, the initial and current reaction rates were determined following the process of numerical differentiation. The kinetic curve was analyzed using the three-point method.

Prior to conducting the experiments, the effect of the amount and degree of granulation of the catalyst on the parameters of the dearomatization process was studied. The results obtained by conducting the experiments at a temperature of 160°C, the Aru: *n*-decene mole ratio (1:0.5), and the reaction time (0.5 h) are presented in Table 4.

Table 4 Effect of the amount and degree of granulation of the catalyst on the rate of n-decene consumption during the process of dearomatization of the kerosene fraction.

	Amount of catalyst, mass%				
	5	10	15	20	
-w, mol/kg-cat·h	12.59	18.45	22.56	22.60	
	The grain size of the catalyst, mm				
	3.5 ÷ 3.0	2.5 ÷ 2.0	1.5 ÷ 1.0	1.0 ÷ 0.64	0.32 ÷ 0.15
-w, mol/kg-cat·h	16.76	19.59	22.09	22.59	22.59

When a minimum of 15.0 wt.% of a catalyst with the grain size in the range of 0.15-1.5 mm is used, the rate of *n*-decene consumption does not depend on the geometric parameters of the granulation and the amount of the catalyst. Therefore, the reaction under study proceeds in the kinetic region.

The effect of the composition of the products obtained during the dearomatization process on the temperature and concentration of *n*-decene is presented in Table 5. It was found that when the temperature and contact time increase, the number of n-decene and aromatic compounds decreases. However, the number of oligomeric products and alkylation products increases under these conditions. It has been reported that the dearomatization products are compounds that are present in the fraction that boils at a temperature of >220°C. The light fraction is primarily composed of monoalkylated aromatic and dimeric products of *n*-decene (average molecular weight: 280), and the heavy fraction contains polymers and oligoalkylated aromatic compounds (average molecular weight: 450).

Table 5 Reaction conditions and results obtained by studying the kinetics of the alkylation process of a kerosene fraction.

№	Temperature, °C	Reaction time, hour	Molar ratio ArH: *n*-decen	Concentration of the components in the initial mixture, mol/L		Composition of dearomatization products for the kerosene fraction, mol/L			
				ArH	n-Decen	ArH	*n*-Decen	I fract.	II fract.
1	160	1	1:0.5	1.33	0.665	0.798	0.377	0.0758	0.048
2			1:0.5	1.33	0.665	0.795	0.299	0.0797	0.075
3			1:1	1.17	1.17	0.630	0.479	0.1074	0.1128
4	160	2	1:2	0.963	1.923	0.464	0.720	0.0684	0.2286
5			1:3	0.815	2.445	0.358	0.831	0.0717	0.2535

6			1:4	0.706	2.825	0.279	0.876	0.0598	0.2813
7	160	3	1:0.5	1.33	0.665	0.759	0.261	0.0947	0.0932
8			1:0.5	1.33	0.665	0.597	0.211	0.0371	0.0837
9			1:1	1.17	1.17	0.466	0.317	0.0407	0.1019
10	180	1	1:2	0.963	1.923	0.343	0.456	0.0466	0.1353
11			1:3	0.815	2.445	0.262	0.482	0.0532	0.1880
12			1:4	0.706	2.825	0.211	0.480	0.0598	0.2131
13			1:0.5	1.33	0.665	0.559	0.199	0.0513	0.1311
14			1:1	1.17	1.17	0.445	0.292	0.0588	0.1756
15	180	2	1:2	0.963	1.923	0.313	0.384	0.0770	0.2558
16			1:3	0.815	2.445	0.253	0.391	0.0709	0.2843
17			1:4	0.706	2.825	0.205	0.424	0.0728	0.3105
18			1:0.5	1.33	0.665	0.532	0.186	0.0505	0.1437
19			1:1	1.17	1.17	0.409	0.269	0.0603	0.1873
20	180	3	1:2	0.963	1.923	0.308	0.365	0.0715	0.2682
21			1:3	0.815	2.445	0.245	0.367	0.0709	0.2928
22			1:4	0.706	2.825	0.191	0.367	0.0652	0.3174
23	200	1	1:0.5	1.33	0.665	0.532	0.239	0.0229	0.0632
24			1:0.5	1.33	0.665	0.439	0.166	0.0261	0.0892
25			1:1	1.17	1.17	0.363	0.257	0.0282	0.1027
26	200	2	1:2	0.963	1.923	0.260	0.365	0.0319	0.1306
27			1:3	0.815	2.445	0.220	0.367	0.0408	0.1710
28			1:4	0.706	2.825	0.191	0.367	0.0414	0.1886
29	200	3	1:0.5	1.33	0.665	0.399	0.146	0.0245	0.1043

Several schemes were proposed to describe the dearomatization process based on the results from the studies conducted on the alkylation of alkylbenzenes by olefins in the presence of solid acid catalysts [9-11].

The Langmuir-Hinschelwood mechanism can describe the interaction between the reagents adsorbed on the Bronsted and Lewis acid centers (1). The Ridyl mechanism can describe the interaction between the olefin-n-decene units adsorbed on the active center and the reagent molecules in the liquid phase (2) and the interaction between the adsorbed aromatic compound with decene in the liquid phase (3).

Analysis of the literature and experimental data obtained allows us to focus on the second scheme of interaction.

Thus, according to the third scheme, only alkylbenzenes should be formed. As for the first scheme, the interaction of two adsorbed particles—a carbonium ion formed during the interaction of an olefin with a Brönsted center of an aluminosilicate and an aromatic compound strongly adsorbed on Lewis acid sites with charge transfer—is also unlikely for electrostatic reasons.

An excess of aromatic compounds for selective alkylation reaction should be used to confirm the results.

The results obtained by studying the interaction between the adsorbed *n*-decene units and the reagents in the solvent volume (*n*-decene and aromatic compounds) agree well with the experimentally obtained results.

Some assumptions must be taken into account while deriving the kinetic equations describing the dearomatization processes: the primary reactions associated with the process of dearomatization are alkylation, oligomerization, and oligoaniline. To simplify the kinetic model, the effects of the realkylation and dealkylation stages are neglected.

Based on the results obtained, the dearomatization of the kerosene fraction following the process of alkylation of aromatic components with *n*-decene can be represented as follows:

$$O + Z \leftrightarrow ZO, \tag{1}$$

$$A + ZO \rightarrow MDA + Z, \tag{2}$$

$$MDA + ZO \rightarrow DDA + Z, \tag{3}$$

$$O + ZO \rightarrow O_2 + Z, \tag{4}$$

$$O_2 + Z \leftrightarrow ZO_2, \tag{5}$$

$$A + ZO_2 \rightarrow AO_2 + Z. \tag{6}$$

where O denotes *n*-decene, Z is the catalyst, ZO denotes the surface compound with an olefin, ZO_2 denotes the surface compound with an olefin dimer, A denotes the aromatic components, MDA denotes the monodecyl aromatic hydrocarbons, DDA represents the- didecyl aromatic hydrocarbons, O_2 represents the- decene dimer, and AO_2 denotes the- oligoalkylate.

The rate of the reactions can be expressed as follows:

$$d\frac{[O]}{d\tau} = -k_1[O][Z] + k_{-1}[ZO] - k_4[O][ZO] \tag{7}$$

$$d\frac{[A]}{d\tau} = -k_2[A][ZO] - k_6[A][ZO_2] \tag{8}$$

$$d\frac{[MDA]}{d\tau} = k_2[A][ZO] - k_3[MDA][ZO] \tag{9}$$

$$d\frac{[DDA]}{d\tau} = k_3[MDA][ZO] \tag{10}$$

$$d\frac{[O_2]}{d\tau} = k_4[O][ZO] - k_5[O_2][Z] + k_{-5}[ZO_2] \tag{11}$$

$$d\frac{[AO_2]}{d\tau} = k_6[A][ZO_2] \tag{12}$$

$$d\frac{[ZO]}{d\tau} = k_1[O][Z] - k_{-1}[ZO] - k_2[A][ZO] - k_3[MDA][ZO] - k_4[O][ZO] \tag{13}$$

$$d\frac{[ZO_2]}{d\tau} = k_5[O_2][Z] - k_{-5}[ZO_2] - k_6[A][ZO_2] \tag{14}$$

As intermediate compounds ZO and ZO_2 are not formed during the reaction (the Bodenstein stationary principle), the derivatives with respect to time should be equal to zero ($d\,[ZO]/d\tau = 0$, $d\,[ZO_2]/d\tau = 0$). In this case, for [ZO] and $[ZO_2]$, we get:

$$[ZO] = \frac{k_1[O][Z]}{k_{-1} + k_2[A] + k_3[MDA] + k_4[O]} \tag{15}$$

$$[ZO_2] = \frac{k_5[O_2][Z]}{k_{-5} + k_6[A]} \tag{16}$$

From the expressions (15) and (16) and the system of equations (7-14), we obtain the following equations for the rate of the chemical reactions:

$$d\frac{[O]}{d\tau} = -\left\{k_1[O] - \frac{\left(1 - {k_4}^1[O]\right){k_p}^1[O]}{1 + {k_2}^1[A] + {k_3}^1[MDA] + {k_4}^1[O]}\right\}[Z] \tag{17}$$

$$d\frac{[A]}{d\tau} = -\left\{\frac{k_2{k_p}^1[O][A]}{1 + {k_2}^1[A] + {k_3}^1[MDA] + {k_4}^1[O]} + \frac{k_6k_5[O_2][A]}{k_{-5} + k_6[A]}\right\}[Z] \tag{18}$$

$$d\frac{[MDA]}{d\tau} = \frac{(k_2[A] - k_3[MDA]){k_p}^1[O][Z]}{1 + {k_2}^1[A] + {k_3}^1[MDA] + {k_4}^1[O]} \tag{19}$$

$$d\frac{[DDA]}{d\tau} = \frac{k_3[MDA][O][Z]}{1 + {k_2}^1[A] + {k_3}^1[MDA] + {k_4}^1[O]} \tag{20}$$

$$d\frac{[O_2]}{d\tau} = \frac{{k_p}^1k_4[O]2[Z]}{1 + {k_2}^1[A] + {k_3}^1[MDA] + {k_4}^1[O]} - \frac{k_5k_6[O_2][A][Z]}{k_{-5} + k_6[A]} \tag{21}$$

$$d\frac{[AO_2]}{d\tau} = \frac{k_5k_6[A][O_2][Z]}{k_{-5} + k_6[A]} \tag{22}$$

where k_p^1 and k_P^5 are the equilibrium constants of the corresponding stages.

$${k_2}^1 = \frac{k_2}{k_{-1}}; \tag{23}$$

$${k_3}^1 = \frac{k_3}{k_{-1}}; \tag{24}$$

$${k_4}^1 = \frac{k_4}{k_{-1}}. \tag{25}$$

The process of cationic oligomerization of *n*-decene was studied, and it was found that the yield of the dimers is significantly lower than the yield of the n-decene trimers under the same conditions. Therefore, we can assume that the first fraction obtained under the conditions of dearomatization primarily consists of mono-alkylated products, and the second fraction consists of oligoalkylates, dialkylated products, and higher n-decene oligomers.

In this case, we obtain the following equations for the reaction rates for the reactions that lead to the formation of products:

$$d\frac{[MDA]}{d\tau}=\frac{(k_2[A]-k_3[MDA])k_p{}^1[O][Z]}{1+k_2{}^1[A]+k_3{}^1[MDA]+k_4{}^1[O]} \tag{26}$$

$$\frac{d([DDA]+[O_2]+[AO_2])}{d\tau}=\frac{\left(k_3[MDA][O]+k_p{}^1k_4[O]^2\right)[Z]}{1+k_2{}^1[A]+k_3{}^1[MDA]+k_4{}^1[O]} \tag{27}$$

Equations (15) and (16) can be rearranged, and the following velocity expressions can be obtained based on the limiting stages of the general reaction mechanism:

1. Assuming that the 1st and 2nd stages are relatively slower than the other stages, and k_{-1} is equal to zero, we obtain the following kinetic equations:

$$\frac{d[MDA]}{d\tau}=\frac{(k_2[A]-k_3[MDA])k_1[O][Z]}{k_2[A]+k_3[MDA]+k_4[O]} \tag{28}$$

$$\frac{d([DDA]+[O_2]+[AO_2])}{d\tau}=\frac{(k_3[MDA][O]+k_1k_4[O]^2)[Z]}{k_2[A]+k_3[MDA]+k_4[O]} \tag{29}$$

2. Assuming that stages 3 and 4 are slow, we obtain the following kinetic equations (provided that $1 + k_2{}^1[A] >> k_3{}^1[MDA] + k_4{}^1[O]$):

$$d\frac{[MDA]}{d\tau}=\frac{(k_2[A]-k_3[MDA])k_p{}^1[O][Z]}{1+k_2{}^1[A]} \tag{30}$$

$$\frac{d([DDA]+[O_2]+[AO_2])}{d\tau}=\frac{\left(k_3[MDA][O]+k_p{}^1k_4[O]^2\right)[Z]}{1+k_2{}^1[A]} \tag{31}$$

3. Assuming that the 1st and 4th stages are relatively slow, we obtain the following kinetic equations (provided that $1 + k_2{}^1[A] + k_3{}^1[MDA] >> k_4{}^1[O]$, a $k_{-1} << 1$):

$$d\frac{[MDA]}{d\tau}=\frac{(k_2[A]-k_3[MDA])k_1[O][Z]}{k_2[A]+k_3[MDA]} \tag{32}$$

$$\frac{d([DDA]+[O_2]+[AO_2])}{d\tau}=\frac{k_1k_4[O]^2[Z]}{k_2[A]+k_3[MDA]} \tag{33}$$

4. Assuming that stage 3 is slow, we obtain the following kinetic equations (provided that $1+ k_2{}^1[A] + k_4{}^1[O] >> k_3{}^1[MDA]$):

$$d\frac{[MDA]}{d\tau}=\frac{(k_2[A]-k_3[MDA])k_p{}^1[O][Z]}{1+k_2{}^1[\mathrm{A}]+k_4{}^1[O]} \tag{34}$$

$$\frac{d([DDA]+[O_2]+[AO_2])}{d\tau}=\frac{\left(k_3[MDA][O]+k_p{}^1k_4[O]^2\right)[Z]}{1+k_2{}^1[\mathrm{A}]+k_4{}^1[O]} \tag{35}$$

Further transformation of the systems of equations (28-35), under the assumptions that $k_{-1} << 1$, $k_1 >> 0$, $k_p^1 >> 0$, $k_{-5} << 1$, results in the generation of the following equations (30, 31):

$$d[MDA]/d\tau = (k_2[A] - k_3[MDA])k_1[O]\,[Z]/k_2[\mathrm{A}]) \tag{36}$$

$$\frac{d([DDA] + [O_2] + [AO_2])}{d\tau} = \frac{k_1 k_4 [O]^2 [Z]}{k_2[A]} \tag{37}$$

The following can be obtained from equation (3.13):

$$d\frac{[MDA]}{d\tau} = \frac{(k_2[A] - k_3[MDA])k_1[O][Z]}{k_2[A] + k_3[MDA]} \tag{38}$$

$$\frac{d([DDA] + [O_2] + [AO_2])}{d\tau} = \frac{k_1 k_4 [O]^2 [Z]}{k_2[A] + k_3[MDA]} \tag{39}$$

The following can be obtained from equation (3.14):

$$d\frac{[MDA]}{d\tau} = \frac{(k_2[A] - k_3[MDA])k_1[O][Z]}{k_2[A] + k_4[O]} \tag{40}$$

$$\frac{d([DDA] + [O_2] + [AO_2])}{d\tau} = \frac{k_1 k_4 [O]^2 [Z]}{k_2[A] + k_4[O]} \tag{41}$$

An analysis of the obtained equations of reaction rates at various limiting stages using computer technology showed that the results obtained from equations 36 and 37 are consistent with the obtained experimental data.

The parameters of the kinetic models were determined by the method of moving tolerance by minimizing the objective function F(x), which characterizes the degree of coincidence of the experimental and calculated data (obtained from the kinetic models). The optimal values are obtained under these conditions.

The dependence of the rate constants of various stages on the reaction temperature (Arrhenius dependence) is presented below:

$$k_1 = 1.45 \cdot 10^7 \cdot \exp\left(-\frac{40018}{\mathrm{RT}}\right) \tag{42}$$

$$k_2 = 1.86 \cdot 10^5 \cdot \exp\left(-\frac{28753}{\mathrm{RT}}\right) \tag{43}$$

$$k_3 = 189 \cdot 10^7 \cdot \exp\left(-\frac{68809}{\mathrm{RT}}\right) \tag{44}$$

$$k_4 = 9.13 \cdot 10^4 \cdot \exp\left(-\frac{29537}{\mathrm{RT}}\right) \tag{45}$$

The obtained kinetic equations satisfactorily describe the experimental data. The average relative deviations of the experimental values from the calculated values are within the acceptable range (10.0%).

Table 6 presents the numerical values of the kinetic parameters corresponding to the stages that affect the rates of alkylation and oligomerization.

Table 6 Numerical values of kinetic parameters.

Constants	Constant values at temperatures 160°C	180°C	200°C	Preexponential factor	Activation energy (kJ/mol)
k_1	214.6	350	549	$1.45 \cdot 10^7$	40.018
k_2	62.96	90	124.4	$1.86 \cdot 10^5$	28.753
k_3	0.093	0.22	0.47	$1.89 \cdot 10^7$	68.809
k_4	24.8	35.7	50	$9.13 \cdot 10^4$	29.537

4. Conclusions

A kinetic model for the process of kerosene dearomatization is proposed. The model is based on the Ridil single-center mechanism, which efficiently describes the process under study. The limiting stages of the process involve the interaction between the *n*-decene unit adsorbed on the surface of the catalyst containing aromatic compounds and the *n*-decene unit present in bulk.

Author Contributions

Statement of the question, selection of goals, processing of results, etc. S. Amirov; conducting experiments, processing results, etc. S. Ismailova

Competing Interests

The authors have declared that no competing interests exist.

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