**Study of the Physico-Chemical and Textural Characteristics of Aromatization Catalysts for C1-C4 Saturated Hydrocarbons**

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**Abstract.** The work studied the physicochemical and textural characteristics of aromatization catalysts for C1-C4 saturated hydrocarbons. Electron microscopy was used to determine the structure and state of the catalyst active centres, and the BET method, which is based on the BET equation, was used to compute the surface area. An XRD-6100 diffractometer (Shimadzu, Japan) was used to examine the catalysts using X-ray (X-ray phase analysis). The generation of coke as a consequence of the catalytic transformations of C1-C4 alkanes was verified by data from IR spectroscopy and electron microscope investigation. Analysis of the IR spectrum of the highly catalytically active catalyst with the (MoO3)x∙(ZnO)y∙(ZrO2)z/HSZ revealed the presence of both ZrO2 components (vibrations in the 1460 and 1140 cm–1 regions) and the MoO3phase (vibrations in the 1000 and 820 cm–1 regions). The research results led to the development of methods for synthesizing HSZ and highly catalytically active catalysts based on them. For the study, a composition of highly catalytically active catalysts with various compositions was synthesized. The concentration of acid centres is contingent upon the extent of decationization of the mesoporous high-silica zeolite, the silicate module of the mesoporous high-silica zeolite, its type, the amount and method of introducing the promoter, and the method of thermochemical treatment.

**Keywords:** Low molecular weight hydrocarbons, aromatization, catalyst, acid center, coke, textural characteristic, IR spectrum, X-ray diffractogram.

**INTRODUCTION**

Recently, due to increasing environmental requirements, the focus in reforming processes has shifted towards producing high-octane gasoline components with a high content of aromatic hydrocarbons (AHCs) or iso-low molecular weight saturated hydrocarbons [1-5]. In petroleum chemistry and industrial oil refining, high-silica mesoporous aluminosilicates serve as selective, highly efficient catalysts for hydrocarbon conversion, especially in the catalytic aromatisation of unbranched normal hexane into aromatic hydrocarbons, primarily benzene, toluene, and xylenes [6-13].

The reforming process combines several main types of reactions. These include the dehydrogenation of six-membered naphthenes, the dehydrocyclization of five-membered naphthenes, and the aromatization (dehydrocyclization) of low molecular weight saturated hydrocarbons, along with chain isomerization. Hydrocracking reactions under hydrogen pressure play a vital part in the reforming process. The most important mechanistic pathways in reforming are considered to be the aromatization of n-low molecular weight paraffins and chain isomerization.

Modern oil refining and petrochemical processes are based on catalytic technologies. Catalytic reformation is a fundamental process in oil refining, facilitating the generation of high-octane motor gasoline components and specific aromatic hydrocarbons such as benzene, toluene, and xylenes [14-19].

Increasing the share of high-octane, benzene-free components (alkylate, isomers, oxygenates) and using environmentally friendly, non-toxic antiknock additives [20] continue to be a focus for scientists worldwide.

To meet the stringent demands placed on oil refining processes, the international market offers the latest catalytic technologies, such as ultra-deep hydrotreating [20-24], isomerization and dehydrogenation processes, as well as hydrogen recycling. The development and modernization of processes, moving away from outdated technologies, are paramount in oil and gas refining. This is driven by increasing quality demands for gasoline as a motor fuel, primarily its octane number.

For modified high-silica mesoporous aluminosilicates, the selectivity for AHCs in n-hexane conversion can increase up to 60%; however, the yield of valuable products in the process decreases significantly. Specifically, in the catalytic aromatization of n-hexane over a selective, highly active catalyst, the amount of propane and methane is approximately 15% each, and ethane is about 5%. The mechanism of aromatization is a very complex process.

In addition to the viewpoints mentioned above, the activation mechanism of low molecular weight saturated hydrocarbon molecules has been proposed, involving either hydride ion abstraction by carbenium ions or initial dehydrogenation, determined by the nature of protonation.

The demand for automotive gasoline and aromatic hydrocarbons is growing daily. Research aimed at improving the process of catalytic aromatization of hydrocarbons for liquid fuel production, thereby increasing the yield of octane-enhancing hydrocarbons, is highly relevant.

**EXPERIMENTAL PART**

**Determination of the structure and surface area of active catalyst centers with high catalytic activity.** The configuration and condition of the active sites of the catalyst exhibiting high catalytic activity were assessed via electron microscopy, while the surface area was evaluated using the BET technique. The BET equation is as follows:

(1)

where P represents the adsorption pressure, measured in Pascals (Pa);

P0 is the saturation pressure, Pa;

V is the adsorption volume, cm3;

Vm is the volume adsorbed in a monomolecular layer, cm3;

C is a constant taken to be equal to a constant characterizing the heat of adsorption.

The specific surface area (S) was determined using the subsequent formula:

(2)

where is the volume of nitrogen necessary to create a monomolecular layer (ascertained from the graph), cm³;

4.38 is the surface area covered by 1 cm3 of nitrogen, m2/cm3;

m is the mass of the catalyst, g.

The equilibrium pressure during adsorption was estimated using the following formula to ascertain the specific surface area via the chromatographic technique:

(3)

where Patm is the atmospheric pressure, mm Hg;

a is the fraction of nitrogen in the nitrogen-helium mixture.

The quantity of adsorbed nitrogen was calculated using the following formula:

(4)

where is the volume of the injected dose, cm³;

is the desorption peak area;

> is the dose peak area.

The adsorption isotherm was constructed based on the relationship between and , where is the saturated vapor pressure of the adsorbate at liquid nitrogen temperature, mm Hg.

The volume of nitrogen adsorbed in a monomolecular layer on the catalyst surface was determined using the following formula:

(5)

where m and b are parameters determined from the graph, 1/cm3.

The relative surface area (Srel) of the catalyst was determined using the following formula:

(6)

where, -1 cm3 adsorbate coverage surface, for nitrogen =4.35m2/cm3;

m1-catalyst mass, g

**X-ray phase analysis:** The catalysts were examined by X-ray diffraction (X-ray phase analysis) using an XRD-6100 diffractometer (Shimadzu, Japan). Phase identification was carried out based on data from the literature by comparing the X-ray diffractograms with those of individual components from the JSPDS (JCPDS) file. The line (peak) intensities were measured on a 100-point scale based on peak heights. The interplanar distance (d, nm) in the crystal lattice was determined using the Wolff-Bragg formula:

(7)

where n is the order of reflection;

λ is the X-ray wavelength, ;

is the Bragg angle, degrees.

The mean crystallite size was determined utilising the Selyakov-Scherrer equation:

(8)

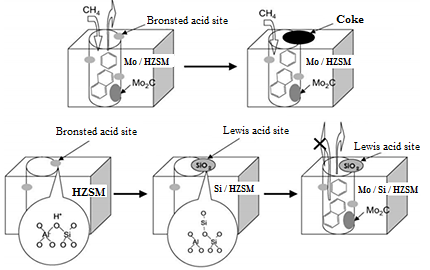
The smaller the particle size of the highly catalytically active catalyst, the lower its diffusion and thermal resistance, and the higher its activity.

**Determining the amount of coke.** The quantity of coke on the highly catalytically active catalysts was ascertained by the mass difference before and after calcination in air. To do this, the highly catalytically active catalyst was subjected to heating in air at 110 °C, and its mass was recorded. Subsequently, it was subjected to a furnace at 800 °C. After one hour, the mass was remeasured, and the quantity of coke was assessed.

**RESULTS AND DISCUSSION**

The primary reasons for the deactivation of highly catalytically active catalysts are their coking (blocking of active sites by coke) and the formation of tarry substances due to the condensation of by-products from the reactions, leading to the blockage of the pores of the highly catalytically active catalyst.

The formation of coke in the layer of a highly catalytically active catalyst can be represented by the following scheme (see Figure 1):



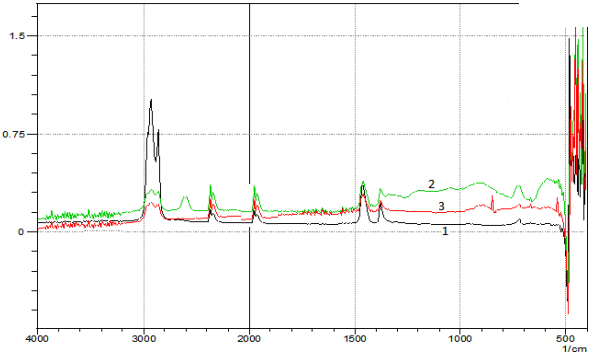
**FIGURE 1.** Scheme of acid site blocking by coke in HZSM.

The amount of hydrogen present in the coke composition is determined by the difference between the amount of oxygen consumed and the amount of carbon dioxide formed.

Thus, this method allows for the determination of the coke structure (graphitized or amorphous). It was found that the error of this method, when compared to results obtained from derivatographic analysis, does not exceed %.

The pore dimensions and specific surface area of the highly catalytically active catalyst influence its propensity for coking. Consequently, the physical adsorption of nitrogen technique was employed to ascertain the specific surface area, along with the dimensions and volume of the catalyst's pores.

Data from IR spectroscopy on coke formation as a result of the catalytic transformations of C1-C4 alkanes were also confirmed by electron microscopy analysis (see Figure 2).



**FIGURE 2.** IR spectra of the coke formed in the reactors with untreated (1), treated with (MoO3)x∙(ZnO)y∙(ZrO2)z (2), and treated with (MoO3)x∙(ZnO)y∙(B2O3)z (3).

The IR spectral analysis of the coke reveals broad absorption bands peaking at 914 cm-1, indicating the development of metal carbides in the coke from reactors whose walls were treated with different salts. The absence of the aforementioned bands, coupled with the existence of disordered carbon structures in the 1350-1500 cm-1 area of the IR spectra, substantiates this observation about coke from reactors with walls coated in various metal salts.

The resultant amorphous coke can be combusted off the reactor surface in an air stream at 550-600 °C over a duration of 2-3 hours.

**Investigating the influence of various factors on the catalytic properties of catalysts.** The acid sites of catalysts for the aromatization of C1-C4 saturated hydrocarbons are a crucial factor determining their catalytic activity. There are two categories of acid sites: weak acid sites and strong acid sites.

Initial studies showed that when using a high-silica mesoporous zeolite derived from bentonite alone for the catalytic aromatization of C1-C4 saturated hydrocarbons at 450°C and a WHSV of 1000 h-1, the formation of aromatic hydrocarbons was not observed. However, when molybdenum metal nanoparticles were introduced into the high-silica mesoporous zeolite from bentonite, the conversion of C1-C4 saturated hydrocarbons into aromatic hydrocarbons was observed.

Initially, zirconium-containing catalysts have a greater number of acid sites compared to those from MoO4**2/**bentonite. In catalytic aromatization reactions, the molar ratios of the catalyst components and the amount of active components are of great importance. This is because these parameters significantly affect the degree of crystallinity of the catalyst. Therefore, we investigated the effect of varying the amounts of promoter added to the catalyst with different ratios of the main catalyst components. The results acquired are displayed in Table 1 below.

**TABLE 1.** The influence of component molar ratios and promoter amounts on the crystallinity degree of the highly active catalyst

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Highly active catalyst** | **Concentration, % mass** | | **Molar ratio** | | **Crystallinity degree, %** |
| **ZrO2** | **Al2O3** | **SiO2/Al2O3** | **SiO2/ZrO2** |
| **1% Zr- HZSM** | 0,9 | 13,5 | 9 | 140 | 100 |
| **2%Zr- HZSM** | 1.8 | 12.3 | 15 | 90 | 95 |
| **3%Zr- HZSM** | 2.0 | 10.6 | 18 | 70 | 94 |
| **4% Zr- HZSM** | 3.2 | 9.2 | 22 | 40 | 80 |

An increase in zirconium content in the highly active catalyst composition results in a decrease in the number of acid sites, hence diminishing its catalytic activity.

Experimental results established that with an increase in the processing duration of high-silica mesoporous zeolites, their acid sites decrease. Table 2 presents the results of studying the effect of temperature and calcination duration on the pore structure.

**TABLE 2.** The surface area and volume of the (MoO3)x∙(ZnO)y∙(ZrO2)z system treated in air at different temperatures

|  |  |  |  |
| --- | --- | --- | --- |
| **Catalyst** | **Thermal treatment conditions** | **Specific surface area, m2/g** | **Pore volume, cm3/g** |
| **HZSM** | 550 0C, 4 h | 338 | 0,88 |
| 600 0C, 24 h | 331 | 0,82 |
| 700 0C, 24 h | 223 | 0,79 |
| **(MoO3)x /** **HZSM** | 550 0C, 4 h | 255 | 0,64 |
| 600 0C, 24 h | 248 | 0,64 |
| 700 0C, 24 h | 247 | 0,63 |
| **(MoO3)x∙(ZnO)y /** **HZSM** | 550 0C, 4 h | 276 | 0,69 |
| 600 0C, 24 h | 274 | 0,67 |
| 700 0C, 24 h | 265 | 0,67 |
| **(MoO3)x∙(ZrO2)z/HZSM** | 550 0C,4 h | 308 | 0,78 |
| 600 0C,24 h | 305 | 0,74 |
| 700 0C, 24 h | 296 | 0,73 |
| **(MoO3)x∙(ZnO)y∙(ZrO2)z/HZSM** | 550 0C,4 h | 291 | 0,69 |
| 600 0C,24 h | 286 | 0,68 |
| 700 0C, 24 h | 282 | 0,66 |

The following Table 3 shows the influence of temperature and duration of thermal treatment on the acid sites of HZSM.

Table 3 indicates that an increase in temperature from 600 °C to 700 °C, along with an extension of thermal treatment duration from 4 hours to 24 hours, results in a monotonous drop in the concentration of both categories of acid sites, predominantly the “strong” acid sites. This is due to the dehydroxylation of the surface of the mesoporous high-silica zeolite, during which two B-sites form one L-site.

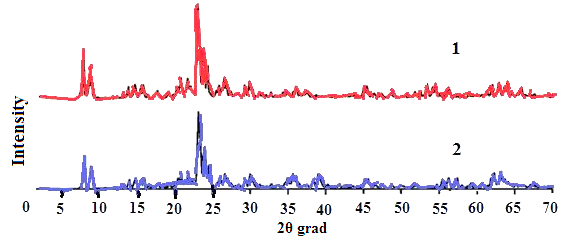
**TABLE 3.** The influence of temperature and duration of thermal treatment on the acid sites of HZSM

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Heat treatment temperature, 0C** | **Heat treatment time, h** | **Amount of desorbed ammonia, μmol/g** | | |
| **100...3500C** | **350...5750C** | **∑100...5750C** |
| 600 | 1 | 598,2 | 312,0 | 989,8 |
| 2 | 579,0 | 352,0 | 986,2 |
| 4 | 556,2 | 321,1 | 784,3 |
| 8 | 528,6 | 300,0 | 733,8 |
| 12 | 508,8 | 256,3 | 715,1 |
| 24 | 492,2 | 200,3 | 662,8 |
| 650 | 1 | 480,3 | 188,8 | 656,7 |
| 2 | 472,3 | 184,9 | 655,8 |
| 4 | 447,4 | 166,5 | 593,6 |
| 8 | 382,6 | 142,8 | 485,8 |
| 12 | 367,3 | 125,6 | 462,3 |
| 24 | 345,2 | 118,1 | 435,3 |
| 700 | 1 | 331,6 | 110,3 | 416,9 |
| 2 | 324,0 | 102,2 | 409,2 |
| 4 | 316,1 | 92,8 | 385,1 |
| 8 | 302,7 | 79,1 | 341,8 |
| 12 | 283,5 | 74,6 | 328,1 |
| 24 | 251,8 | 61,8 | 272,7 |

Consequently, the experimental results indicate that the optimal temperature for thermal treatment is 600 °C.

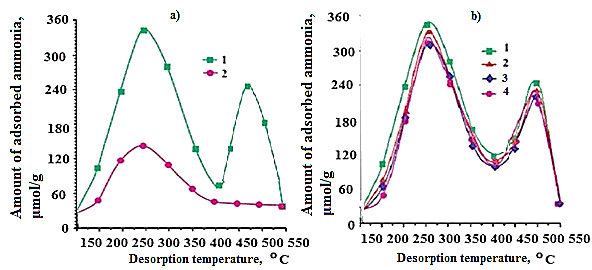
To determine the phase composition of the highly active catalyst with the proposed composition (MoO3)x(ZnO)y(ZrO2)z(B2O3)k/HZSM, the catalyst was analyzed by X-ray diffractometry both before and after the reaction. The results obtained from the X-ray diffractometry are presented in Figure 3.

The positions of the characteristic peaks in the diffractograms presented in Figure 3, obtained by X-ray diffractometry, are typical for the highly active catalyst with the composition (MoO3)x(ZnO)y(ZrO2)z(B2O3)k/HZSM. The desilicated (MoO3)x(ZnO)y(ZrO2)z(B2O3)k/HZSM catalyst (see Figure 3) retains its structure; however, the intensity of the peaks characteristic of the (MoO3)x(ZnO)y(ZrO2)z(B2O3)k/HZSM catalyst decreases, which is associated with a partial degradation of the catalyst structure.



**FIGURE 3.** Diffractograms of MoO3)x(ZnO)y(ZrO2)z(B2O3)k/HZSM before (1) and after (2) the experiment

The acidic properties of the surface of highly catalytically active catalysts were studied using the ammonia TPD (Temperature-Programmed Desorption) method. The longer the thermal treatment duration of the catalysts, the more the “strong” acid sites are destroyed, and the concentration of acid sites decreases. The relationship between the thermal treatment duration of the highly catalytically active catalyst and the concentration of acid sites is presented in Figure 4 below.



**FIGURE 4.** Ammonia desorption curves from the surface of HZSM and (MoO3)x∙(ZnO)y∙(ZrO2)z /HZSM calcined in air at 550°C: a) 1 - HZSM; 2 - -(MoO3)x∙(ZnO)y∙(ZrO2)z /HZSM (thermal treatment duration 4 hours). b) (MoO3)x∙(ZnO)y∙(ZrO2)z /HZSM (thermal treatment duration: 1 - 4 hours; 2 - 24 hours; 3 - 36 hours; 4 - 48 hours).

**CONCLUSION**

Methods for synthesizing the initial reagents, HZSM, and highly active catalysts based on them were studied, and catalyst compositions with various structures were synthesized for the research.

The influence of acid sites on the activity of the catalytic system in the hydrocarbon aromatization reaction was determined. In turn, the dependence of the concentration of acid sites on numerous factors was investigated, including: the degree of decationization of the mesoporous high-silica zeolite, the silicate module of the mesoporous high-silica zeolite, its type, the amount and method of introducing the promoter, and the method of thermochemical treatment.

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