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Mathematical Modeling of Changes in the Fractional Composition of Dehydrogenation Catalysts in a Fluidized-Bed Reactor

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Abstract—A mathematical model describing the destruction of catalyst particles during operation in an industrial fluidized-bed reactor with allowance for the crushing and abrasion of particles is proposed. Differences between the mechanical properties of IM-2201 and KDI catalysts used in the dehydrogenation of *iso*-butane to *iso*-butylene at PAO Nizhnekamskneftekhim are established. Using KDI catalyst with modification of the cyclone group and retention of the equilibrium distribution of particles according to size in a reactor is shown to provide a more than a 2.5-fold reduction in catalyst consumption.

Keywords: mathematical modeling, chromia–alumina catalyst, dehydrogenation, grain, fractional composition, crushing, abrasion

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INTRODUCTION

The dehydrogenation of C₄–C₅ *iso*-paraffins to *iso*-olefins under industrial conditions is usually performed in chromia–alumina catalyst fluidized bed reactors. Catalyst particles strongly collide with each other and reactor walls during operation, leading to their crushing and abrasion. Changes in the fractional composition of a catalyst due to the destruction of its grains during operation must be taken into account when designing a reactor, since this can be of critical importance.

The crushing and abrasion of grains depend on many factors, particularly, the strength of particles and the design features of a reactor. Numerical modeling and experimental studies of the crushing of discrete solid phase particles were considered in [1–9]. However, these works did not consider the crushing and abrasion of grains in combination, and the proposed mathematical models were in a sophisticated form and required special parametric implementation for each type of catalyst. The last factor made their direct application to the problem considered in this work problematic.

The first part of this work considers the simplest mathematical model describing the dynamics of particle sizes in a fluidized-bed reactor. The model takes into account the two basic processes in the destruction of catalyst grains, crushing and abrasion. The second part of this work presents some results from laboratory experiments demonstrating the possibility of adapting this model to IM-2201 and KDI dehydrogenation catalysts, the ones most in demand for the production of

iso-butylene at PAO Nizhnekamskneftekhim. The third part of this work shows the applicability of the developed mathematical model to modeling the operation of actual industrial reactors.

MATHEMATICAL MODEL OF THE CRUSHING AND ABRASION OF GRAINS

General Description of the Process

Any model describing the dynamics of changes in the fractional composition of catalyst particles in a fluidized-bed reactor must take into account the two basic processes leading to changes in their size, crushing and abrasion. The first of these processes is quite fast. Its characteristic time is several hours, much shorter than the time catalyst particles typically spend in a reactor and regenerator until the complete replacement of the catalyst in the system. We may therefore consider the process of crushing to be infinitely fast and describe it with a crushing matrix. This matrix allows us to calculate the effective fractional composition after the particle crushing stage from the fractional composition of the catalyst loaded into the reactor. The crushing matrix depends on the type of a catalyst and must be found using the results from laboratory experiments. The abrasion of particles is a much slower process. The characteristic time of abrasion is close to the time particles spend in a reactor and regenerator until the complete replacement of the catalyst in the system. Any description of the dynamics of a population of catalyst particles must therefore take

into account the detailed kinetics of their abrasion, in contrast to crushing. The kinetic model of the abrasion of particles is limited to a transfer equation, in which time and particle size are independent variables, and the particle size distribution density is the sought function.

Crushing of Grains

Let us separate each fraction loaded into a reactor (initially or during the process) into two components. The first of these corresponds to brittle and easily destructible particles that break into smaller grains a short time after their loading into a reactor. We ignore this short (relative to the characteristic time the particles spend in a reactor) time when characterizing the infinitely fast process of initial crushing by transition matrix D with dimensions $N \times N$. Here, N is the number of different fractions numbered according to their sizes from 1 (the coarsest fraction) to N (the finest fraction). Elements D_{kn} of matrix D characterize the mass content of particles from the k th fraction formed upon the crushing of particles from the n th fraction. The following conditions must then be met:

$$d_{kn} = 0 \quad (k < n), \quad \sum_{n=1}^k d_{kn} = 1. \quad (1)$$

These indicate that (1) coarse particles cannot be formed from fine particles as a result of crushing, and (2) the total mass of fragments is equal to the mass of a crushed particle. Equality $d_{NN} = 1$ following from Eq. (1) means that the particles formed upon the crushing of grains from the finest fraction remain in the same fraction.

Matrix D can and must be determined as a result of experiments on the dynamics of the abrasion of grains in the initial period of time. The crushing of particles in a reactor during operation is ignored after its termination, as was mentioned above. Crushing is accounted for by recalculating the fractional composition of the catalyst initially loaded and effectively loaded (after crushing) into the reactor, using matrix D . If the mass of the k th fraction initially loaded into a reactor is denoted as m_k , the effective distribution $m^0 = (m_1^0, m_2^0, \dots, m_N^0)^T$ of fractions according to size after the infinitely fast stage of grain crushing is found as the product of matrix D and vector $m = (m_1, m_2, \dots, m_N)^T$:

$$m^0 = Dm.$$

The fractional composition of the catalyst additionally loaded into a reactor during operation is recalculated in the same fashion. Let us denote the column vectors of the real and effective additional catalyst loading rates by fractions as q and q^0 (kg/s), respectively. Then

$$q^0 = Dq.$$

Abrasion of Grains

Let us denote the mass of the catalyst in a reactor at specific moment in time as M (kg) and the mass of the catalyst added into a reactor and unloaded from it per unit time as Q_+ and Q_- (kg/s). We refer to the finest fraction formed during the abrasion of particles as dust. Let us denote the mass contents of dust inside a reactor and in the catalyst added into a reactor and unloaded from it as φ , φ_+ , and φ_- , respectively. The maximum size of dust-forming particles is denoted as a_{dust} . The size distribution of particles in the region of $a > a_{\text{dust}}$ inside a reactor and at its inlet and outlet are characterized by mass densities $f(a)$, $f_+(a)$, and $f_-(a)$ (1/m), respectively. By definition, $f(a)da$ is the mass of particles in a reactor with sizes within the range $[a, a + da]$ per unit mass of all particles in the reactor. The values of $f_{\pm}(a)$ can be found in the same fashion. By definition,

$$\int_{a_{\text{dust}}}^{\infty} f(a)da = 1 - \varphi, \quad \int_{a_{\text{dust}}}^{\infty} f_{\pm}(a)da = 1 - \varphi_{\pm}. \quad (2)$$

Let us consider the balance of dust for time dt . Dust mass increment $d(M\varphi)$ in the system is a result of adding dust at the inlet of the system ($Q_+\varphi_+dt$), withdrawing dust from the system ($Q_-\varphi_-dt$), and the abrasion of particles. The abrasion of particles with sizes $a > a_{\text{dust}}$ is characterized by rate of abrasion v (kg/kg/s), defined as the mass of dust formed per unit time from the unit mass of particles with size a . Dust due to abrasion is added into the system for two reasons. The first of these is the mass loss of particles with sizes $a > a_{\text{dust}}$. Its corresponding value is $M \left(\int_{a_{\text{dust}}}^{\infty} v(a)f(a)da \right) dt$. The second reason is the reduced size of particles. The mass of a particle is proportional to its cubed size, so a particle with size a shrinks in time dt by $da = \frac{1}{3}av(a)dt$. As a result, particles with sizes in the range $[a_{\text{dust}}, a_{\text{dust}} + da_{\text{dust}}]$ also become dust, yielding a dust quantity increment of $\frac{1}{3}Ma_{\text{dust}}v(a_{\text{dust}})f(a_{\text{dust}})dt$. The mass balance of dust in a reactor is described by the relation

$$\begin{aligned} \frac{d}{dt}(M\varphi) = & M \int_{a_{\text{dust}}}^{\infty} v(a)f(a,t)da \\ & + \frac{1}{3}Ma_{\text{dust}}v(a_{\text{dust}})f(a_{\text{dust}},t) + Q_+\varphi_+ - Q_-\varphi_-. \end{aligned} \quad (3)$$

Writing the mass balance of particles with sizes in the range $[a, a + da]$ in a similar fashion, we obtain

$$\begin{aligned} \frac{d}{dt}(M\varphi) &= -Mv(a)f(a,t) \\ &+ \frac{1}{3}M\frac{\partial}{\partial a}(avf) + Q_+f_+ - Q_-f_- \end{aligned} \quad (4)$$

Let us integrate both parts of this equation over a within the range from a_{dust} to infinity using Eqs. (2), and sum the result with Eq. (3). We obtain the integral equation of the catalyst mass balance,

$$\frac{dM}{dt} = Q_+ - Q_-, \quad (5)$$

expressing the obvious fact that the catalyst mass in a reactor can change only as a result of the addition and withdrawal of particles, and not due to their abrasion. Dust balance equation (3) is fully equivalent to catalyst mass balance equation (5) and can be replaced with it. As a result, we arrive at Eqs. (4) and (5) for calculating the sought functions $M(t)$ and $f(a,t)$. At specified values of $Q_{\pm}(t)$, the catalyst mass in the system is found via the simple integration of Eq. (5). After $M(t)$ is calculated, the distribution density is found by solving transfer equation (4), and quantity $\varphi(t)$ of dust in the system is determined by the first of Eqs. (2).

To close Eq. (4), we must specify dependence $v(a)$ of the rate of abrasion on particle size. This dependence can vary for different types of catalysts. Laboratory experiments are the most direct method for measuring $v(a)$. We use the same method below.

EXPERIMENTAL

Laboratory Experiments

Our laboratory experiments to estimate mechanical strength were performed for two types of catalysts, KDI and IM-2201. Five fractions of each catalyst with sizes of 100–125 (fraction 1), 70–100 (fraction 2), 40–70 (fraction 3), 20–40 (fraction 4), and $<20 \mu\text{m}$ (fraction 5) were separated for the tests. Each of the first three fractions (individually) and fractions 4 and 5 (together) were loaded into the vessel of an abrasion strength testing unit (Fig. 1). Hence, four experiments were performed for each of the two catalysts. Abrasion occurred under the action of an air jet passed through a nozzle in the lower section of the vessel for 24 h. The fractional composition of a loaded sample was determined 3, 6, 15, and 24 h from the beginning of each experiment. A typical pattern of the changes in fractional composition is shown in Fig. 2 using the KDI catalyst as an example.

Adapting the Mathematical Model to Fit Laboratory Data

Under experimental conditions, $Q_+ = Q_- = 0$, $M = \text{const}$, and Eq. (4) after dividing by M is transformed into the equation

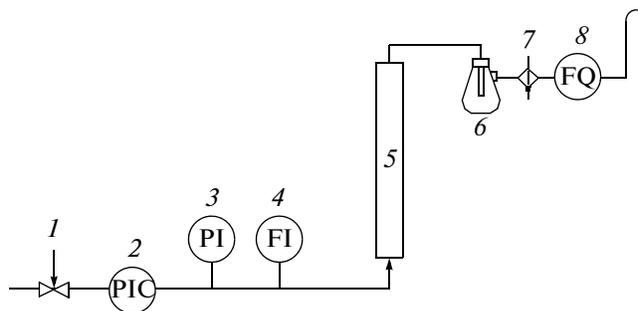


Fig. 1. Schematic of the unit for estimating the mechanical strength of catalysts in an air jet: (1) valve, (2) SDV-6 pressure stabilizer, (3) manometer, (4) rotameter, (5) abrasion vessel, (6) coarse filter, (7) fine filter, (8) gas counter.

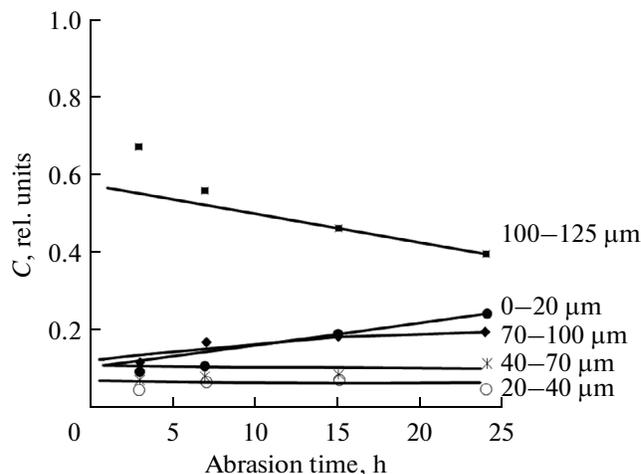


Fig. 2. Concentration of different KDI catalyst fractions versus abrasion time in our experiment with initial fraction 1 (points) and in calculations (lines).

$$\frac{df}{dt} = -v(a)f(a,t) + \frac{1}{3}\frac{\partial}{\partial a}(avf). \quad (6)$$

The solution to Eq. (6) was found by an explicit scheme: Eq. (6) was discretized over a uniform grid with step h within the range $a_{\text{dust}} < a < a_{\text{max}}$. The typical number of discretization nodes N was assumed to be 256. The computational scheme was

$$\begin{aligned} \frac{\hat{f}_i - f_i}{\tau} &= -v_i f_i + \frac{1}{3h}(a_{i+1}v_{i+1}f_{i+1} - a_i v_i f_i) \\ i &= N-1, N-2, \dots, 1, \end{aligned} \quad (7)$$

where \hat{f} is the distribution density at the next time level, and τ is the time step.

After \hat{f} was found using explicit formula (7), the mass content of fractions 1–4 were determined via numerical integration of \hat{f} over the corresponding ranges, and the mass content of dust (fraction 5) was calculated using Eq. (2). The true mass contents of the fractions at the initial moment in time were calculated

from the content of the fractions loaded into the reactor using crushing matrix D . Dependence $v(a)$ was initially selected to be a piecewise-constant function that assumed different values within each fractional range.

In processing the experimental data, we considered that the abrasion of the coarsest fraction in each experiment did not depend on the finer fractions, so the dynamics in the k th experiment was completely governed by two parameters, rate of abrasion v_k assigned to the corresponding fractional range, and coefficient D_{kk} of its crushing. The two coefficients v_k and D_{kk} can be uniquely determined from the content of the coarsest fraction measured in a mixture at two points (15 and 24 h). Rates of abrasion v_k found in this fashion for both catalysts in different time periods proved to be close and differed from one another by no more than 10%. This allowed us to further assume that rate of abrasion v was constant and independent of the particle size. We determined it for the KDI and IM-2201 catalysts as

$$v_{\text{KDI}} = 0.0065 \text{ h}^{-1}, \quad v_{\text{IM}} = 0.0115 \text{ h}^{-1}.$$

The other ten model parameters (coefficients $D_{21}, D_{31}, D_{32}, D_{41}, D_{42}, D_{43}, D_{51}, D_{52}, D_{53}, D_{54}$ of matrix D) were determined in a manner to ensure the best agreement between the theoretical and remaining ten experimental curves. As a result, we found the crushing matrices

$$D_{\text{KDI}} = \begin{pmatrix} 0.59 & 0 & 0 & 0 & 0 \\ 0.12 & 0.73 & 0 & 0 & 0 \\ 0.11 & 0.12 & 0.91 & 0 & 0 \\ 0.07 & 0.10 & 0.09 & 1 & 0 \\ 0.11 & 0.05 & 0 & 0 & 1 \end{pmatrix}, \quad (8)$$

$$D_{\text{IM}} = \begin{pmatrix} 0.25 & 0 & 0 & 0 & 0 \\ 0.1 & 0.78 & 0 & 0 & 0 \\ 0.17 & 0.17 & 0.92 & 0 & 0 \\ 0.19 & 0.05 & 0.03 & 0.95 & 0 \\ 0.29 & 0 & 0.05 & 0.05 & 1 \end{pmatrix}.$$

The quality of agreement between theory and experiment for the KDI catalyst is illustrated in Fig. 2. It would seem to be quite satisfactory. The mathematical model adapted for use with laboratory tests was therefore also used in computational experiments to estimate the equilibrium fractional compositions of a catalyst in actual reactors.

Let us now turn to the differences between the tested KDI and IM-2201 catalysts. First of all, the rate of IM-2201 catalyst abrasion was nearly twice that of the KDI catalyst. Second, the coarse IM-2201 grains were much more brittle than the KDI grains. This difference is explained by the type of cohesion between the primary particles of the catalysts, the natures of which were similar and determined by their phase and

chemical composition [10, 11]. The IM-2201 catalyst was synthesized using spray drying technology, where coarse agglomerates are formed during synthesis due to weak coagulation contacts between primary particles. The grains of KDI catalyst synthesized by impregnating a high-strength support with active component solutions are formed due to the strong crystallization bonds between primary particles. According to [8], this is why only 25% of the coarse IM-2201 catalyst grains remain after flash crushing, while their content for the KDI catalyst is only 60% (the upper left elements of the crushing matrices). At the same time, the finer grains of both catalysts are comparable in brittleness [8].

EQUILIBRIUM DISTRIBUTION OF PARTICLES IN AN INDUSTRIAL REACTOR

Formulation of the Problem and Its Solution

From a practical viewpoint, it is important to know the equilibrium distribution of particles in an industrial reactor according to size. Let us clarify what this means:

Let relatively coarse particles determined by distribution function f_+ be added into a reactor at constant flow rate Q_+ , while small catalyst particles are withdrawn from it at the same flow rate ($Q_- = Q_+$). The total catalyst mass in the system remains constant, and a steady-state (equilibrium) distribution $f(a)$ of particles according to size is attained in the system over time. The main aim of this work is to determine such a distribution.

Before moving to our mathematical formulation of the problem, let us note several important factors:

(1) The fractional composition of a catalyst in a reactor is usually measured in respect to the same fractions used in laboratory experiments. We may therefore use crushing matrix D measured in experiments to describe processes that occur in industrial reactors;

(2) Rates of abrasion v for particles in the laboratory and in industry can vary due to the differences between the intensities of processes and the designs of reactors. Since the jet velocity at the outlet of a nozzle is 225 m/s under laboratory conditions and 22.5 m/s under industrial conditions, and the catalyst remains in a laboratory unit until abrasion is complete and periodically enters the zone of slow velocities and a dense phase (e.g., in the lower section of a reactor) in an industrial unit, it is obvious that the intensity of particle collisions in a laboratory reactor is much higher. The rate of abrasion would therefore be expected to slow considerably when describing an industrial reactor, compared to the rate of abrasion measured in a laboratory. The ratio between laboratory and industrial rates of abrasion must nevertheless remain the same for all types of catalysts; and

(3) Particles of the finest fraction ($<20 \mu\text{m}$) generally penetrate the existing dust collecting system of an

Table 1. Fractional composition of IM-2201 catalyst, wt %

| Catalyst | Fraction, μm | | | | |
|---------------------------|-------------------------|-------|-------|--------|------|
| | <20 | 20–40 | 40–70 | 70–100 | >100 |
| Added | 6.5 | 9.6 | 20.1 | 18.1 | 45.7 |
| Equilibrium (actual) | 0 | 29.2 | 36.7 | 19.5 | 14.6 |
| Equilibrium (theoretical) | 0 | 28.9 | 41.8 | 16.3 | 13.0 |

industrial reactor (a group of cyclones). In addition, an appreciable amount of 20–40- μm particles (up to 15 wt %) is observed at the outlet of a reactor. It is therefore convenient to fix size a_{dust} of dust removed from a reactor in the range of $20 < a_{\text{dust}} < 40$ when selecting the corresponding value from the conditions of best agreement with the industrial data on the distribution of particles inside a reactor and at the outlet from it.

In light of these assumptions, let us finalize our formulation of the problem of a uniform distribution of catalyst particles inside a reactor, based on the above mathematical model. We introduce dimensionless parameter $V = Q_+/Mv$ equal to the ratio between the rate, at which a catalyst is fed into a reactor and the rate of its abrasion. In accordance with Eqs. (4) and (2), equilibrium distribution $f(a)$ is described by the common differential equation

$$-f + \frac{1}{3} \frac{\partial}{\partial a} (af) + Vf_+(a) = 0, \quad a_{\text{dust}} < a < \infty \quad (9)$$

and the integral condition

$$\int_{a_{\text{dust}}}^{\infty} f(a) da = 1, \quad (10)$$

which reflects all of the dust (particles with radii $a < a_{\text{dust}}$) being removed from the system. Additional condition (10) helps us calculate dimensionless catalyst feed parameter V , at which an equilibrium distribution of particles according to size is attained in a reactor. Let us recall that effective distribution f_+ of particles at the inlet of a reactor in Eq. (9) is recalculated via the true (effective) distribution using crushing matrix D .

Since f is directly proportional to V [9], there is no problem in solving (9), (10). At each fixed $f_+(a)$ and a_{dust} , it determines the required catalyst feed parameter V and equilibrium distribution $f(a)$ of particles in a reactor. It can also be used to calculate the content of particles with sizes in the range of $20 < a < a_{\text{dust}}$ (C_{20-40}) in catalyst withdrawn from a reactor, using the formula

$$C_{20-40} = \frac{1}{3} a_{\text{dust}} f(a_{\text{dust}}) + \int_{20}^{a_{\text{dust}}} f_+(a) da. \quad (11)$$

It is obvious that the content of finer withdrawn particles is $C_{0-20} = 1 - C_{20-40}$.

Analyzing Industrial Data

Until recently, IM-2201 catalyst was generally used in the production of *iso*-butylene at PAO Nizhnekamsk-neftekhim. A wide spectrum of industrial data whose statistical processing allowed us to determine the average fractional composition of catalyst added into a reactor (upper row of Table 1), the average equilibrium composition of catalyst in a reactor (second row), and the percentage of coarse (more than 20- μm) fractions entrained through cyclones from a reactor, was accumulated over the period of its use. This percentage was 16% on average, and was used to find the model's only adaptation parameter: the lower size limit for the particles removed from a reactor, $a_{\text{dust}} = 25 \mu\text{m}$. The equilibrium composition of the catalyst in a reactor (third row in Table 1) and parameter V , proven to be 1.6%, were then determined by solving problem (9), (10). As can be seen from Table 1, the actual and calculated equilibrium fractional compositions were in good agreement with one another. Knowing the catalyst volume (300 t) and the fresh catalyst flow rate (8 t/day), we can determine the universal coefficient of 0.0065 for moving from laboratory to industrial rates of abrasion. This means the rate of abrasion in an industrial reactor is 160 times lower than in the laboratory.

A mixture of IM-2201 (60%) and KDI (40%) is now frequently used for the additional loading of fresh catalyst into a reactor. The fractional composition is given in Tables 1 and 2. When such a mixture is used, the withdrawal of coarse (more than 20- μm) fraction through cyclones grows considerably, reaching 28%. The model thus requires a_{dust} to be raised from 25 to 34 μm . The actual fractional composition of the mixture in a reactor and its fractional composition calculated with such a_{dust} (second and third rows in Table 2) are in satisfactory agreement with one another.

Due to the higher abrasion of IM-2201 catalyst, the equilibrium ratio between the catalysts in a reactor differs considerably from the ratio at the moment of loading. The KDI content in the equilibrium composition is thus already 65%. Dimensionless catalyst feeding rate V proves to be 1.2, meaning a 25% reduction in catalyst consumption.

Table 2. Fractional composition of a mixture of the IM-2201 and KDI catalysts (60 : 40), wt %

| Catalyst | Fraction, μm | | | | |
|---------------------------|-------------------------|-------|-------|--------|------|
| | <20 | 20–40 | 40–70 | 70–100 | >100 |
| Added KDI | 0.5 | 11.5 | 28.0 | 16.0 | 44.0 |
| Equilibrium (actual) | 0 | 12.0 | 42.0 | 32.0 | 14.0 |
| Equilibrium (theoretical) | 0 | 13.0 | 45.7 | 25.3 | 16.0 |

A high volume of 20–40- μm particles withdrawn from the reactor—and, as a consequence, a low content of such particles in the equilibrium composition (13%)—are observed due to the unsatisfactory operation of the cyclone group. Upgrading it could substantially improve these results, even for a reactor operating only with KDI catalyst. As an example, let us consider such a modification of cyclones that yields $a_{\text{dust}} = 25 \mu\text{m}$ for both KDI and IM-2201 catalysts. In this case, loading KDI with the fractional composition from the first row of Table 2 produces the following equilibrium composition: 20–40 μm , 25.8%; 40–70 μm , 38.5%; 70–10 μm , 20.1%; >100 μm , 14.6%, almost reproducing the equilibrium composition of IM-2201 catalyst (see Table 1). The withdrawal of the coarse fraction remains almost the same at 16.3%, while the dimensionless fresh catalyst feed parameter falls from 1.6 for IM-2201 to 0.63 for KDI.

CONCLUSIONS

(1) A mathematical model of the destruction of IM-2201 and KDI catalyst particles in the fluidized beds of industrial reactors for the dehydrogenation of *iso*-paraffins to *iso*-olefins has been proposed and adapted using laboratory data. The model considers both the fast crushing of particles and the slow process of their abrasion.

(2) The proposed model describes the available data on the operation of industrial flow-type reactors with a fluidized catalyst bed.

(3) When analyzing the crushing matrix, it was established that the crushing matrix coefficients for KDI coarse fractions are 2.5 times lower than the corresponding coefficients for IM-2201.

(4) It was established that using KDI catalyst in combination with the modification of the cyclone group can reduce catalyst consumption by more than 2.5 times at the same equilibrium distribution of particles in a reactor according to size.

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