Irada Arif Khalafova
Azerbaijan State Oil and Industry University
Baku, Azerbaijan

Zivyar Islam Ismailova
Azerbaijan State Oil and Industry University
Baku, Azerbaijan
kerem_shixaliyev@mail.ru

INTENSIFICATION OF THE CATALYTIC CRACKING PROCESS UNDER THE INFLUENCE OF A MAGNETIC FIELD

Abstract: The aim of this work is to study the effect of a magnetic field on the fluidized bed hydrodynamics of an industrial catalytic cracking catalyst. In article, we investigated the use of pretreatment of vacuum gas oil, its mixtures with fuel oil and a process catalyst in the catalytic cracking process, and also considered the problem of the effect of a constant magnetic field both on the conversion of heavy oil feedstock and on the hydrodynamics and structure of the catalyst itself. The processing of oil and oil products by a magnetic field is used as one of the technological processes that allow, for example, to reduce the growth rate of paraffin deposits, accelerate the separation of oil and water, reduce the viscosity of oil, etc. The physicochemical mechanisms of the action of the magnetic field on a number of processes in oil and gas production, and in particular on the deep refining of oil in the process of catalytic cracking, is based on the assumption of the presence in the processed solutions of impurities of colloidal ferromagnetic iron particles, which exist in the form of aggregates, including the participation of other colloidal particles present in the solution.

Key words: pressure drop, accelerator, magnetic field, fluidized layer, critical speed, inductance, hydrodynamics, pressure losses, zeolite-containing catalyst, selectivity, vacuum gas oil.

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Introduction
Research part:

When interacting with the magnetic field of ferromagnetic particles, the destruction of aggregates occurs, which later manifests itself in the form of changes in the course of physicochemical processes [1].

It turned out that in liquids, ferromagnetic iron particles exist mainly in the form of rod-shaped crystals with a length of less than 1 micron (10^{-6} m), and these microcrystals are combined into structures - aggregates of sufficiently large sizes, in which thousands of such particles are combined. Under the influence of a magnetic field, the aggregates are crushed into fragments, which are smaller aggregates and individual particles, which change the properties of solutions. The fragmentation of aggregates leads, first of all, to a manifold increase in the amount of solid particles, which serve as centers of crystallization and gas formation. In a sufficiently strong external magnetic field, an aggregate including a gas bubble and a ferromagnetic particle will collapse, since its constituent particles acquire the same orientation along the field and, due to the repulsive forces, move away from each other. After a while, if the particles did not participate in other chemical and physical processes, the aggregates are formed again. Consequently, aggregates of ferromagnetic particles of iron oxides are an object that interacts with a magnetic field and exhibits

numerous secondary effects of magnetic treatment [2].

Method

The magnetization of ferromagnet colloids is carried out by two independent mechanisms [3]. The first of them is the Néel process, known for supermagnetic solid dispersions, of overcoming by a magnetic moment under the influence of thermal fluctuations of energy barriers separating its equilibrium position in a particle. The second is the process of magnetization by orienting the magnetic moment together with a particle in a viscous medium. The latter mechanism is observed only in magnetic colloids if the intensity of the magnetizing field H is less than the characteristic intensity of the effective anisotropy field H_2, which fixes the magnetic moment along the selected directions in the particle. Since, depending on the material of the particles H_2, it is on the order of $10^2 - 10^3$ Oe, it is necessary to take into account both mechanisms of magnetization.

According to the results of [5], the transition of iron particles with a Curie temperature of 500–1000 K from the ferromagnetic to the superparamagnetic state, in which ferromagnetism disappears due to the disordering of the magnetic moments, occurs when the particle size is $D \leq 24$ nm in diameter. By definition, the point T_c on the temperature scale that separates the regions of the disordered paramagnetic phase $(T > T_c)$ and the ordered ferromagnetic phase $(T < T_c)$ is called the Curie point. According to [6], the Curie temperature of ferromagnetic particles when their size decreases to 2 nm does not differ from T_c bulk metals.

In the course of studying the properties of colloids of natural oils (both at the micro and nanolevels), it was found [7] that any oil is a colloidal system, in which the colloidal phase is represented mainly by asphaltenes, which have the ability to self-associate into a large number of clearly different (nano) colloidal structures in a richly structured phase diagram. Associative or "micellar" colloids, as a rule, have a wide phase diversity: from the simplest isotropic, micellar phases to complex supermolecular nanostructures [8].

As shown in [9], the reason for the emergence and existence of sediment called asphaltenes is that paramagnetic molecules exist in oil and petroleum products or are formed in homolytic reactions. Homolytic reactions are characterized by the breaking of an electron pair; for example, redox reactions proceed according to this type. These paramagnetic molecules are rejected from the medium used as a solvent molecules containing σ-bonded atoms (for example, saturated hydrocarbons) into a precipitate. The latter is a concentrate of paramagnetic molecules containing an admixture of coprecipitated diamagnetic molecules (with a well-developed π system of bonds, metal complexes, heterosystems).

Without paramagnetic molecules, asphaltenes do not exist and asphaltenes cannot be non-paramagnetic. The total number of paramagnetic molecules grows with time with increasing temperature and pressure. Paramagnetism of asphaltenes initially increases, but with the appearance of carbenes - carbides, it stabilizes regardless of temperature and pressure. Carbene-carbides, despite the increase in their number over time, have specific paramagnetism, which does not depend on temperature, pressure, time, i.e. constant for each type of raw material. The appearance of carbenes-carbides corresponds to saturation of the specific paramagnetism curve of asphaltenes.

Analysis of numerous data from various instrumental methods and their comparison [10] indicate that heavy carbon molecules or individual carbon atoms combine into large structures such as associates, polymers, or crystal lattices due to spin excitation and spin polarization through the influence of a solvent and (or) temperature. Asphaltenes, a paramagnetic powder rejected by molecules with zero spin polarization, are a reflection of such processes. Thus, not only asphaltenes, but also heavier and less soluble sediments of oil dispersed systems are of a purely paramagnetic nature.

Experimental studies have established [10] that reversible singlet-triplet transitions are inherent in one way or another to all oil-like systems. Under the action of a magnetic field, new radicals or biradicals are formed due to the singlet-triplet transition. The magnetic effect of singlet-triplet transitions $(S-T)$ generates chemical polarization of electrons (CPE) and nuclei (CPN). Spin polarization and magnetic effects in radical reactions are two closely interrelated phenomena based on a common physical mechanism [11].

Gasoline fractions have a paramagnetic impurity at the level of 10^{15} cn/g. It can be deposited with a large excess of saturated hydrocarbons $C_4 - C_5$, but the precipitate is not a powder, but a viscous-resinous substance. The same kind of impurity of resins and paramagnets in one or another amount is present in all petroleum products. The planted resins have different paramagnetism and, with its large value, a low-intensity spectrum of nuclear magnetic resonance (NMR). A similar pattern is observed for asphaltenes. The lower their paramagnetism, the more resolved and more intense the NMR spectrum.

The paramagnetism of petroleum products and petroleum-like substances, estimated by the number of paramagnetic centers (PMC), varies from 10^{15} spin/g for the gasoline fraction to 10^{22} for calcined cokes. Asphaltenes or more condensed compounds that make up the core of the dispersed particle have stable paramagnetic characteristics. The paramagnetic characteristics of residual products of distillation of gas condensate and oil were studied [12]: the registered amount of PMC varied from $6 \cdot 10^{17}$ to $5 \cdot 10^{18}$ depending on the nature of the oil product. For

all samples, the greatest effect from magnetic exposure was observed at the lowest speed: the increase in paramagnetic activity was 38-57%. A decrease in the amount of PMC in oil residues a few hours after exposure to a magnetic field turns out to be a process of radical recombination, which agrees with the data in [13].

A paramagnetic sample placed in a constant magnetic field can absorb the energy of an electromagnetic field applied to it. The phenomenon of electron paramagnetic resonance, which consists in the absorption of electromagnetic radiation by paramagnetic samples has a selective (resonant) character, since it is observed only at a certain ratio between the strength of the constant magnetic field and the frequency of the alternating electromagnetic field. In the absence of an external magnetic field $\vec{H}$, the chaotic thermal motion of paramagnetic particles leads to averaging to zero of the total magnetic moment $\vec{M} = 0$. If the sample is placed in a constant magnetic field $\vec{H}$, the magnetic moments of the paramagnetic particles are oriented along the direction of the vector $\vec{H}$; therefore, a nonzero total magnetic moment appears, i.e. the sample is magnetized. The higher the magnetic field strength $\vec{H}$, the more the sample is magnetized. In relatively weak magnetic fields, the magnitude of the induced magnetic moment: $\vec{M} = \chi\vec{H}$, where is χ - the magnetic susceptibility (usually, $\chi = 10^{-3} - 10^{-6}$). Paramagnets have a magnetic moment $\vec{M} \neq 0$ and are oriented in the direction of the external field.

The specific features of the magnetic properties of nanoparticles are associated with the discreteness of their electronic and phonon states. Magnetization is determined by the difference between the magnetic field induction in the sample and the external magnetic field induction, and the magnetic susceptibility χ is determined as the ratio of magnetization to the external magnetic field strength [15].

As the results of experimental and theoretical studies [16-20] show, the use of magnetic processing can significantly increase the efficiency of the processing of petroleum feedstock. It was found that when a magnetic field is applied to hydrocarbon and water systems, the decisive role is played by: the magnitude of the magnetic induction, the rate of intersection of the magnetic field in the active zone (zone with maximum induction) by the flow of the processed liquid, and the number of intersections of the magnetic field by the processed liquid.

From this point of view, the intensification of the processing of heavy oil feedstock using physical methods of influencing the oil system is an urgent problem.

A fluidized (fluidized) bed is formed when a gas (liquid) passes through a bed of a finely dispersed catalyst (contact) at a certain speed, at which the channels formed between the bed particles and penetrating it in all directions expand and the bed volume increases. This leads to mixing and movement of particles relative to each other, and the layers as a whole exert hydrostatic pressure on the walls of the apparatus.

The fluidized bed method is a progressive method for the technological processing of various types of raw materials. This is explained by the fact that when using a fluidized bed, maximum contact between the raw material and the heat carrier is ensured, without which chemical, physical and thermal processes cannot effectively proceed. The greater the dispersion of the solid phase forming the fluidized bed, the larger the contact surface of the reactants and the more efficiently the process proceeds. Another valuable feature of fluidized bed processes is the comparative simplicity of technological schemes and equipment, which is explained by the simplicity of the process of separating the reaction products from the bulk of the dispersed solid phase and withdrawing it from the reactor into a regenerator for heating and reactivation.

Results

The advantages of using a fluidized bed for contact coking of heavy oil refining wastes are especially prominently revealed. In this case, the particles of the fluidized bed are carriers of coke from the reactor to the regenerator and heat from the regenerator to the reactor. In the process of contact coking of heavy feedstock with a fluidized bed of coke oven coolant, a large amount of coke is formed on it, part of which is burned out in the regenerator to enter the required amount of heat into the reactor, and part of it is removed from the system. At the same time, the amount of coke oven fluid in the system is kept constant.

From the analysis of the essence and mechanism of the fluidized bed, it follows that the main disadvantages of fluidized bed systems that reduce the efficiency of contact catalytic heterogeneous processes in such systems are [22]:

1) the breakthrough of a significant part of the reacting substances through the thickness of the layer in the form of bubbles;

2) intensive mixing of fine particles in the bed, leading to equalization of the catalyst activity in the entire volume of the bed to a level equal to the activity of the spent catalyst leaving the apparatus;

3) partial mixing of the reactants and reaction products in the volume of the fluidized bed, which is caused by intensive mixing of the solid phase, and part of the substance is mainly mixed passing through the thin channels between the catalyst particles. This to some extent reduces the reaction rate of this part of the feedstock and promotes the secondary conversion of the reaction products;

4) lower relative speed of phase movement compared to the fixed layer and, as a consequence, the presence in some cases of external diffusion deceleration of the process speed.

The presence of the listed disadvantages does not allow to fully use the potential capabilities of the principle of contacting fine particles with gases and vapors. Therefore, in recent years, the efforts of many researchers have been directed to find ways to improve systems with a fluidized bed in order to increase the efficiency of the processes carried out.

In order to study the effect of a constant magnetic field (MF) on the fluidization hydrodynamics of zeolite-containing cracking catalysts (Omnikat-210P), we studied a catalyst fraction with a particle size of 65-80 mkm and a bulk density of 0.65 g/cm^9.

Below in table 1 shows the dependences of the ΔP gradient on the flow rate of the fluidizing agent (air) on the W catalyst without the influence of MF, and in table 2 shows the values of the pressure loss ΔP from the gas supply rate W (m/s) and the calculated dependences W between the hydraulic resistance ΔP and the air supply rate in the absence of a magnetic field, as well as the values of the Reynolds number Re, which characterizes the ratio of inertia and viscosity forces in the flow and, accordingly, the type of flow - laminar or turbulent. It was determined that at $Re_{kr} > 10$, there is a transition from the filtration mode to the fluidized bed mode.

Then the effect of the MF on the hydrodynamics of the "fluidized bed" of a finely dispersed catalyst was studied. In this case, the reactor with the catalyst was placed in a MF with an inductance of 0.25-0.30 T.

Table 1. Dependence of the pressure drop on the flow rate of the fluidizing agent and the hydraulic resistance of the layer in the absence of the MF

Layer state	Diffmamometer indicator		Rotameter №1	Catalyst bed height, mm	Rotameter №2	Time in minutes	V (dm^3, l) gas meter	Air temperature, $^O C$
	Right	Left						
Filtration mode	-8	+1	0	19,8	0	65,44	3	18
The beginning of the appearance of bubbles in the layer	-11	+1	0	19,8	0	30,20	3	18,5
The beginning of the fluidized bed	-11	+1		20,0	0	25,33	3	18,5
Fluidized bed	-11	+1	15	21,0	0	22,24	5	19
Fluidized bed	-11	+1	20	22,0	0	20,10	5	19,5
Fluidized bed	-12	+2	78	23,5	0	10,56	5	19,5
Fluidized bed	-13	+3	90	23,5	10	9,01	5	19,5
Fluidized bed	-13	+3	100	24,0	20	8,12	5	20
Normal layer (bubbles in the layer)	-14	+1	0	20,0	0	18,06	3	20
Fluidized bed	-14	+1	10	22,0	0	15,07	3	20
Fluidized bed	-14	+2	20	25,0	10	10,40	3,5	20

Table 2. Hydrodynamics of "fluidized bed" catalyst without MF

Calculations of the experimental part				
Criterion Re	ΔP (mm wg)	Lg ΔP	W (m/s)	LgW
2,4	9	0,845	$1,3 \cdot 10^{-3}$	-2,89
5,1	12	1,079	$2,79 \cdot 10^{-3}$	-2,55

6,086	12	1,079	$3,32 \cdot 10^{-3}$	-2,478
11,055	12	1,079	$6,03 \cdot 10^{-3}$	-2,22
12,8	12	1,079	$7 \cdot 10^{-3}$	-2,15
23,8	14	1,146	$13 \cdot 10^{-3}$	-1,89
51,3	16	1,204	$28 \cdot 10^{-3}$	-1,55
31,7	16	1,204	$17,3 \cdot 10^{-3}$	-1,76

In fig. 1 shows the curves of fluidization of the catalyst (Omnikat-210P) in the absence of MF and under the influence of MF with a strength of 0.25-0.30T.
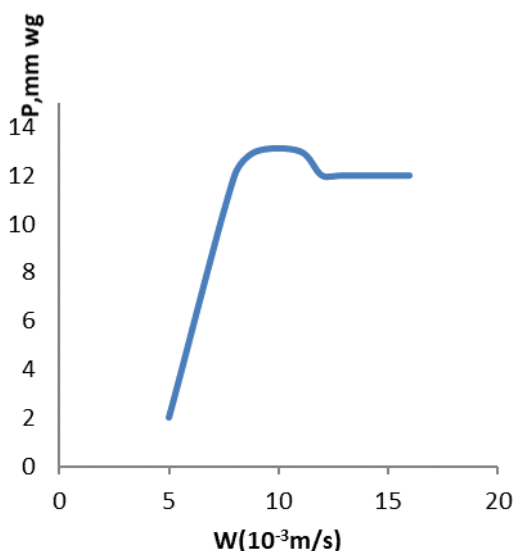
a)
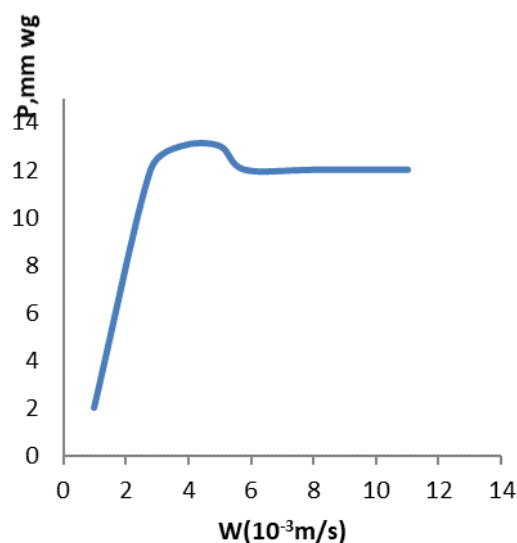
b)

Fig. 1. Catalyst fluidization curve:
a - without the influence of the MF; b - for magnetically treated catalyst

As can be seen from Fig. 1.a, and the relationship between the pressure loss ΔP on the air flow rate at low speeds up W to a certain level, from point 1 to point 2, changes according to a linear law. At point 2, the air flow rate will be the critical filtration rate W_{kr}, then the increase in speed depends entirely on the contact between the bed particles. In the zone between points 2 and 3, an increase in the layer volume by 5-10% is observed, in other words, a decrease in density and a violation of its homogeneity. The beginning of "fluidization" of the bed begins at an air flow rate of 10 m/s. Between points 3 and 4, a "fluidized" bed regime is observed. At the same time, an increase in the air flow rate does not lead to an increase in the loss ΔP, but, on the contrary, to its decrease, and only after point 4 it ceases to be dependent on the air supply rate - the particle hovering mode begins after point 7. From fig. 1.a, tab. 1 and 2, it can be seen that the stabilized laminar regime passes to the fluidized one with an increase in the average flow velocity and is accompanied by a change in the visual pattern of the flow.

A slightly different picture was observed in the case of using a magnetically treated catalyst. The transition from the filtration mode (points 1-2) to the fluidization mode (points 5-7) is observed at a lower W_{kr} critical value of the agent feed rate.

Fig.1.b it can be seen that the magnetic field 2 times accelerates the achievement of the transition period from the filtration mode to the fluidization mode. If the fluidization regime for the initial catalyst began at an air flow rate of $W = 10$ m/s, then for a magnetically treated catalyst this state was observed at a speed of $W = 5$ m/s.

Visual observations also showed that the mixing of the particles of the gas-catalyst flow is more efficient in the presence of MF, and at the same time realizes a uniform fluidized bed, which is characterized by the same concentration of solid particles throughout the volume of the bed and its constancy in the flow and in time. Consequently, the

MP affects the flow regime, namely, the tendency to accelerate the transition from the filtration regime to the transient regime, increases the quality of the fluidized bed, converting it to the uniform, while homogenization of the flow leads to an increase in the yield of target products, an increase in selectivity and productivity catalyst.

As is known [22], an uneven fluidized bed that forms when fine particles come into contact with gases or vapors is characteristic of almost all contact catalytic heterogeneous processes. It is characterized by the passage of gas through the layer by channels (channeling) and bubbles (bubbling), depending on the size of fine particles.

A uniform fluidized bed is formed when a liquid is used as a fluidizing agent and is not typical for catalytic heterogeneous processes carried out by contacting fine particles with gases and vapors. However, the effect of MF on the catalyst, as shown by calculations and visual observations, leads to the formation of a uniform fluidized bed and, as a consequence, to a more intense contact between the feedstock and the catalyst, leading to an increase in the yield of gross products [23-25].

There are the following types of fluidized bed: a) homogeneous; b) with bubbling of gas bubbles; c) with channeling; d) with powder formation; e) with gushing.

The studies carried out allow us to conclude that the effect of MF on the cracking catalyst leads to an acceleration of the transition from the filtration mode to the fluidized one, and in this case, a predominantly homogeneous fluidized bed (type a) is realized, which is stable throughout its existence, which, in turn, it contributes to an increase in the yield of the target reaction products, an increase in the selectivity of the process, etc.

The effect of MF on the composition and structure of the catalyst was also confirmed by physicochemical studies of catalyst samples before and after MF treatment by X-ray fluorescence microscopy (GT-7000, Horiba) and diffuse reflection on a SPECORD M40 spectrophotometer.

X-ray fluorescence microscopy data showed that the magnetic field changed the distribution of elements in the catalyst composition over the depth of the layer. Elemental analysis of the investigated catalyst (Omnikat-210P) was carried out. Samples were copied to determine the chemical composition and penetration depth of the beam. Analysis of the chemical composition of the presented samples shows the presence of 5 main component: $Al_2O_3, SiO, Ti, Fe_2O_3, La_2O_3$. At an accelerating voltage (50 kV), the measurement time was 200s. Analysis of the obtained layer-by-layer spectrum of these elements shows a change in the depth of fluorescence radiation for La and Al on the order of 20-30% higher relative to the initial catalyst (wt %). All this indicates that the structures of the phase states of the catalyst change in the course of magnetic treatment. In fig.2 shows the spectra of the elemental composition of the Omnikat-210 catalyst samples without and after treatment with a magnetic field.
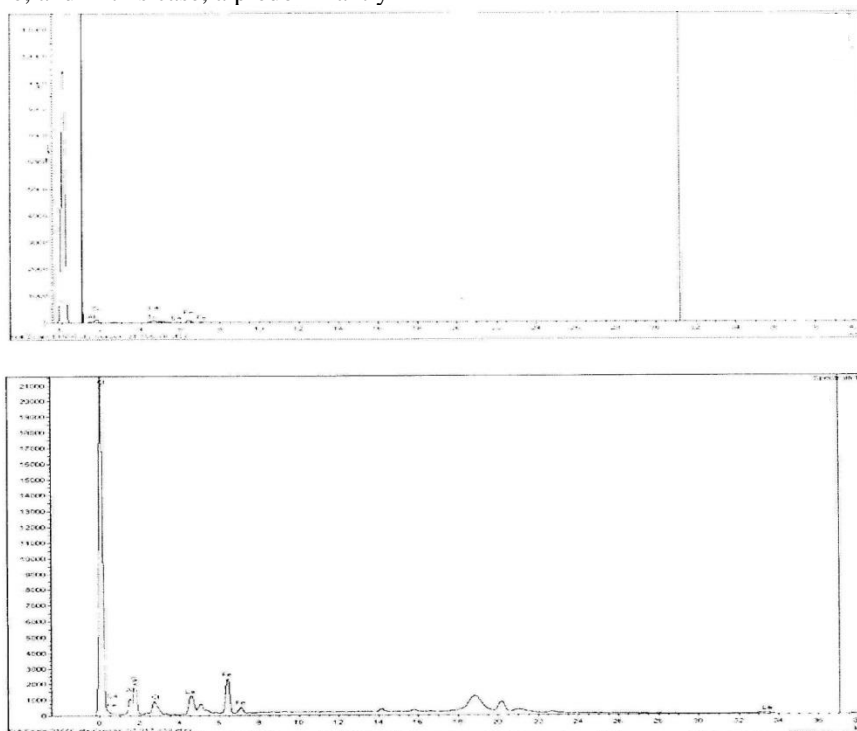

Fig.2. Spectra of the elemental composition of the Omnikat-210P catalyst samples:
a - without magnetic field treatment; b - after treatment with a magnetic field

We also recorded electronic diffusion reflectance spectra (EDRS) in the range of 30000-10000 sm^{-1} on a SPECORD M40 spectrophotometer for the initial catalyst sample and the sample treated with MF. It was found that the EDRS spectrum of the initial sample is characterized by structureless absorption, covering almost the entire investigated spectral region. Due to the absence of clear absorption bands (a.b.), this spectrum does not allow one to unambiguously determine the state of iron ions.

The spectrum of the treated catalyst differs significantly from the original one. Thus against the background of broad absorption, a a.b. at 25500 and 20000 sm^{-1}. Observed a.b. in the spectra of the treated catalyst at 25500 and 20000 sm^{-1} can be attributed to the absorption of Fe^{2+} and Fe^{3+} ions in octahedral coordination, which are stabilized in the system after the catalyst is treated with a magnetic field.

Result:

Thus, the study of the hydrodynamics of the fluidized bed of a finely dispersed catalyst showed that the magnetic field accelerates the achievement of the transition period from the filtration mode to the fluidization mode at a lower flow rate of the fluidizing agent, improves the quality of the fluidized bed, converting it into a uniform one.

In this case, more efficient mixing of the particles of the gas-catalyst flow leads to an increase in the yield of target products, an increase in the selectivity and productivity of the catalyst.

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