TEKA. COMMISSION OF MOTORIZATION AND ENERGETICS IN AGRICULTURE – 2013, Vol. 13, No.3, 56-62

Using the aerosol of SiO_2/ZrO_2 catalyst nanoparticles to obtain gasoline and diesel from vacuum gasoil

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Received June 14.2013: accepted July 08.2013

Summary. It has been examined the use of aerosol nanocatalysis technology for implementing new high catalytic cracking process to produce gasoline and diesel fuel from a vacuum gas oil. the temperature in the new process is at 250 °C lower than in industrial cracking processes, and the amount of catalyst is reduced to a concentration of 2.38 g/m^3 reactor. The SiO_2/ZrO_2-catalyst at conditions of the new technology displayed higher than 99 % of selectivity of light products formation.

Key words: gasoline, diesel, aerosol nanocatalysis

INTRODUCTION

Catalytic cracking currently is the most important and large-tonnaged process among other oil refining processes.

The overall productive capacity of catalytic cracking units in USA is higher than 250 million tons/year of the raw materials. This process has gained a very wide development in the countries of Western Europe. The total capacity of catalytic cracking units has reached 35% in U.S. (13.9% in Western Europe, and 6.0% in Russia) of the capacity of primary oil refining [5,12,18].

In the 60s of the past century, it was discovered the high activity of zeolites in cracking reactions. In this regard, in cracking process they started to use catalysts with Y- zeolite content ($Me_{2/n}O \cdot Al_2O_3 \cdot xSiO_2 \cdot yH_2O$) not more than 20% wt, which resulted in a significant growth of the yield of desired products of the process, especially at the transition to the cracking of heavy feedstocks.

In order to utilize from all advantages of zeolite catalyst, they began to use new types of reactor-regenerator devices: at first was the fluidized bed reactor, and then the lift-reactor. Thus, the process of catalytic cracking became the fastest growing in the oil refining. Gasoline and diesel fuel in the rural economy needs [4, 17].

At present, the work is continuing to intensify the process of catalytic cracking based on the modernization of the reactor and regenerator of catalytic cracking units, simplifying the construction of reactor-regenerator block, creating the most effective ways of contacting the catalyst with the raw materials and preparation of raw materials in order to increase the yield of desired products and improve their quality [22]. But at the same time from the intensifications of existing technologies topically remains the task of creating new ways of carrying out a catalytic cracking process with increased technical and economic indicators.

In the Technological Institute of East-Ukrainian National University, Volodymyr Dahl (Severodonetsk) at the Department of Technology of organic substances, fuels, and polymers are conducted perspective researches on the study of cracking of vacuum gasoil by using the aerosolized nanocatalysis (AnC). The essence of aerosolized nanocatalysis technology is to apply a continuous mechano-chemical activation of the catalyst directly in the reaction volume, resulting in decreasing the required amount of catalyst to 1 - 2 g/m^3 reactor and increasing the catalyst activity up to 10^5 - 10^6 times.

Researches of properties of different catalysts were already conducted (Nexus-345p, Y-zeolite, Si/Zr-catalyst) at conditions of AnC, they showed the possibility of increasing the yield of light products in the 1.14 times, reducing the volume of rector up to 10 times and the volume of regenerator to 1500 times in comparison with the existing technologies [10].

This work is a logical continuation of research in this field, and its main purpose is to study the kinetic characteristics of a new modification of Si/Zr-catalyst at conditions of aerosolized nanocatalysis with the subsequent creating of the basics of technology of catalytic cracking with increased technical and economic indicators.

ANALYSIS OF PUBLICATION, MATERIALS, METHODS

The disadvantages of the industrial organization of catalytic cracking process, which greatly increase the capital and operating costs include [7, 16]:

1. In order to obtain a productive capacity of 2 million tones/year of raw material (unit G-43-107/M1), the volume of reactor (zone of separation and reaction volume) is 800 m^3, the volume of regenerator - 1600 m^3;
2. The need of constant regeneration of catalyst after the 1 - 2 seconds of work, which leads to additional losses of the catalyst due to its abrasion. In the industry this requires an additional makeup of catalyst in an amount of 0.545 kg per ton of raw material, which is 3 tons of additional catalyst in 24 hours, with a minimum price of catalyst ≈ 25 thousands U.S.$/ton;
3. The relatively high concentration of catalyst (up to 700 kg/m^3 reactor);
4. The relatively low specific productivity of reactor and catalyst (205.3 $kg/(m^3_{reactor}.hr)$ and 0.444 $kg/(kg_{kat}.h)$).
5. Necessity of steam injection into the reaction zone for stripping the cracking products from the catalyst surface in an amount of 0.5 - 0.75 GJ/ton raw material;
6. The Relatively big single charge of catalyst in the reactor (350 tons).

At the end of the twentieth century, a new trend in the technology of implementing gas-phased chemical processes has been formulated by Ukrainian scientists - aerosol nanocatalysis (AnC - Aerosol nanoCatalysis) [6]. Its distinctive features:

- catalytic system consists of moving dispersing material at a size of 1 - 2 mm, and a powder of catalyst with initial size till 200 microns;
- the internal diffusion stages are excluded from the catalytic process;
- synthesis (in situ) of superactive nanoparticles of catalyst at a size of 8-100 nm;
- avoiding the use of carrier (catalyst and dispersing material in the reaction space are not integrated in a homogeneous system);
- implementing (in situ) of a continuous mechano-chemical activation on the surface of catalyst by moving solid material;
- equal access of reactants on the active surface.

It was experimentally shown and theoretically proved that the reaction speed rises at about 10^4 - 10^6 times in comparison with traditional catalysis on carriers. This can be explained by the characteristic properties of the nanoparticles surface, which can be remained for 15-30 seconds [1, 11]. Content of catalyst in the reaction zone is reduced to 1-5 g/m^3 of the reaction volume [15]. (under the classical catalysis on carriers of catalyst mass , the catalyst concentration is 0.7- 0.8 ton/m^3 and more, including catalytically active material on basis of noble metals - up to 10 kg or 200 - 1000 kg of other substances).

In reactors of aerosol nanocatalysis, the catalytically active material undergoes a continuous mechano-chemical activation (in situ) by means of forced mechanical oscillations of an inert dispersing material - (glass spheres with diameter of 1.0 - 1.2 mm). A continuous grinding of the coagulated particles of catalyst is applying and converting the particles to nano-sizes (10^{-8} - 10^{-9} m) and maintaining their high activity for unlimited time [20].

Thus the vibro- fluidized bed reactor allows to regulate the frequency of pulses of mechano-chemical activation of particles surface. The surface of nanoparticles is full of free valence bonds, electrons, and structural defects [21]. These factors determine the effectiveness of AnC.

Currently, there are two technologies of research and possible equipment design of the aerosol nanocatalysis in the industry. The first suggested technology is with fluidized bed (ACFB-Aerosol Catalysis with Fluidized Bed) of inert particles with approaching to ideal displacement by regime of movement of reagents and catalyst aerosol. At present, the technology was fulfilled on a number of large experimental units (reactors with diameter of 150 - 250 mm and a height of up to 3 m) with a recycle of catalyst, when developing ACFB it must be considered the resistance of layer of inert particles. The possible time of contact was limited by the speeds range of beginning the fluidizing and carrying over the solid phase, which constricts the technology possibilities.

Recently a relatively new proposed technology of AnC (AnCVB - Aerosol nanoCatalysis with Vibrating Bed) with vibro-fluidized bed of catalytic system. The mechanical activation of the catalyst is applied by forced vibration of reactor. Regime of motion of reactants in the reactor is approaching to the ideal mixing. It is possible to organize the regime to be closer to the displacement. The technology is free from the characteristic disadvantages of ACFB :

The layer has a very low resistance and can be operated at any speeds of the gas stream. Furthermore, for some processes the recirculation of catalyst can be excluded. The catalyst is constantly undergoing to mechano-chemical activation and displaying a catalytic activity in 10 - 100 times higher than in the option ACFB, i.e. 10^5 – 10^6 times higher than the achieved activity in the catalysis on carriers [9]. However, the organization is required vibrating layer with certain parameters. Working with literature and our own experience have shown significant benefits of using vibrated reactor AnC, especially for kinetic studies [19]. In principle, in a new and extremely effective it was showed that it is possible by changing the frequency and amplitude of vibration to control by the regime of mechano-chemical activation and optimize the conditions for each individual taken reaction. Managing of vibration allows to apply in principle a new organization of chemical reactions and extends the possibility of controlling by process. Furthermore, the regime of mechanical activation changes the catalyst activity AnC.

Set of specific properties AnCVB [16] allows to anticipate in advance the improving of indicators of the existing production and to increase the effectiveness of the catalytic cracking (CC) of oilproducts.

1. Increasing the reaction speed and corresponding reduction of the residence time of raw material in the reaction zone due to the exclusion of internal diffusion stages of the process at aerosol nanocatalysis technology.
2. Using metal oxides as cracking catalysts in amounts of 0.3 - 10 g/m^3 reactor instead of large volumes of existing industrial catalysts on carriers with additives of noble metals. This will reduce the capital and operating costs and decrease product cost.
3. In pyrolysis process, using these catalysts will lower the process temperature and reduce energy costs.
4. Technology of aerosol nanocatalysis will constantly maintain a high activity of catalyst. The produced coke will not be fixed on the active surface of the catalyst particles since the nanoparticles do not have pores and they subject to continuous mechano-chemical activation; consequently, will be possible to exclude (or substantially reduce) the steam flow into the reaction zone since the suppression of coke formation reactions will not be required.
5. Technology of aerosol nanocatalysis on vibrating bed has no restrictions on the residence time of material in the reaction zone, that will allow to approach the products output, which is closer to the thermodynamically possible.

Technology AnC will allow to principally change the flow chart of cracking processes, work with a small mass of catalyst and catalyst makeup will be no more than 5% or without catalyst makeup. Apparently, it is possible to exclude stages of regeneration and recirculation of catalyst from the technical scheme of the process. it only needs to arrange a separation of catalyst particles and coke from the reaction products , coke oxidation and supplying the thermal energy to cracking zone . In the industry, these solutions are realized, it is reasonable for AnC [8]. All this will lead to reduce capital and operating expenditures during processing of oilproducts.

The industrial catalyst Nexus - 345p (Grey Davidson, England) was the first catalyst, which was studied for the catalytic cracking process in the conditions of AnCVB. The results obtained from him exceeded the industrial indicators, as follows, [2, 3, 13, 14]:

1. to increase the total yield of the gasoline and diesel fractions in 1.14 times;
2. reduce the reactor dimensions (volume) up to 420 m^3;
3. reduce the total loaded charge of catalyst from 350 ton to 70 kg;
4. the catalyst concentration became only 1 g/m^3 of the reactor (less than in the industry);
5. owing to the small amount of the used catalyst, it is possible to avoid the regeneration stage or make it periodical;
6. the specific productivity of reactor and catalyst increased to (1184 kg / (m$^3_{reacter}$ .h) and 394548 kg / (kg$_{kat.}$. h)) respectively;
7. avoiding use of steam , since there are no pores on the catalyst at conditions of AnCVB ;

therefore the reaction products will not clog them [10].

For this catalyst the temperature of process was 570-640^{o}C at conditions of AnCVB.

PURPOSE AND DIRECTION OF RESEARCH TASKS

Purpose - evaluating effectiveness of creating the necessary conditions for process of catalytic cracking of vacuum gasoil using technology AnCVB.

Tasks of the experimental part of study:

- learning the procedure of conducting the experiment;
- modernize the laboratory unit with a set of the placed experimental tasks;
- learning the procedure of obtaining the initial raw material;
- learning the procedure of analytical control;
- learning the procedure of preparing the catalyst;
- obtaining preliminary results of studies;
- determining the optimal parameters of conducting the process and studying their influence;
- evaluate the perspective of continuing the work in this direction.

THE EXPERIMENTAL PART AND RESULTS DISCUSSION

Process of catalytic cracking at conditions of AnCVB was conducted on laboratory unit shown on fig 1. [13, 20].

The heated vacuum gas by a syringe batcher (1) is fed into the reactor (5) which is located inside a thermal locker (2), and heated by heater (13). In the reactor, the cracking reactions take place. The temperature in the reaction zone is measured by thermocouple (11) and supported by a regulator (12). Metal-clothed filter is provided in order that the catalyst will not be carried away from the reaction zone along with the cracking products (6). The products after the reactor pass through water-cooled condenser and enter into the receiver of liquid fraction (8), the uncondensed gases of cracking pass a point of samples selection (9), and enter into gas washer (10), where the gases are bubbled through the water layer and then enter into the receiver of gaseous phase (15) by passing through water vessel (14). The reactor performs reflexive and translational motions with assistance of vibrating device (4), frequency of oscillation is provided by regulator (12).

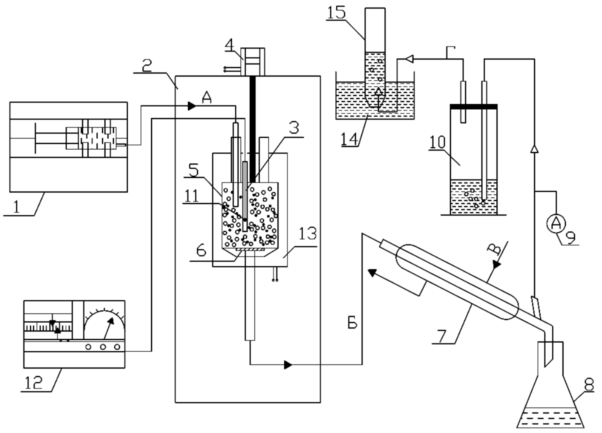

Fig. 1. Laboratory unit for studying process of cracking of vacuum gasoil at technology of aerosol nanocatalysis

1 – syringe batcher; 2 – thermal locker; 3– pocket for thermocouple; 4 – vibrating device; 5 – reactor; 6 – metal-clothed filter; 7 – water-cooled condenser; 8 – receiver of liquid fraction; 9 –point of gathering samples for analysis; 10 – gas washer; 11 – thermocouple; 12 – regulator for oscillation frequency and temperature;13 – heater; 14 – vessel with water; 15 – collector of gaseous phase

Experiments were conducted at temperatures 300^0C, 350^0C, 400^0C, 450^0C, 500^0C, 550^0C, and at frequency MCA 4 - 7 Hz .

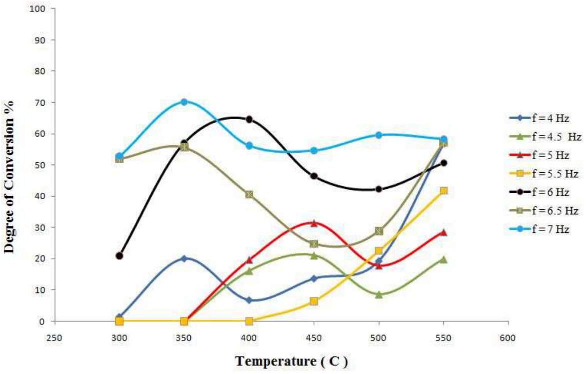

Fig. 2. Graphic of degree of conversion (% weight) vs. temperature (^{0}C)

The maximal degree of conversion (more than 70% weight) for the studied interval of parameters was observed at 350^0C and frequency MCA 7 Hz (fig.2). With further growing of temperature, the degree of conversion decreases in a little. At frequency of 6 Hz, the maximal conversion of gasoil (more than 64%) was observed at 400^0C. It may be noted that the maximal values of degree of conversion based on temperature are observed almost for all the studied range of activation frequency. Perhaps, this is due to the fact that at low temperatures there is intensive MCA on the catalyst surface, which then becomes less intensive because of the thermal softening of catalyst.

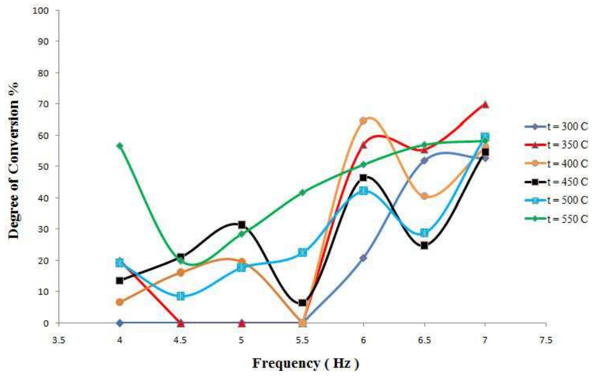

Fig. 3. Graphic degree of conversion (% weight) vs. frequency of vibration (Hz)

At frequency range MCA of 4 - 5.5 Hz and temperature 300-350°C, degree of conversion was 0%, with a gradual increase of frequency to 6 Hz , the degree of conversion increased to 64.6% at 400°C and then to 70% at 7 Hz and a temperature of 350°C (fig. 3). It is obvious that the catalyst has become more active by increasing the frequency of MCA, even at low temperatures.

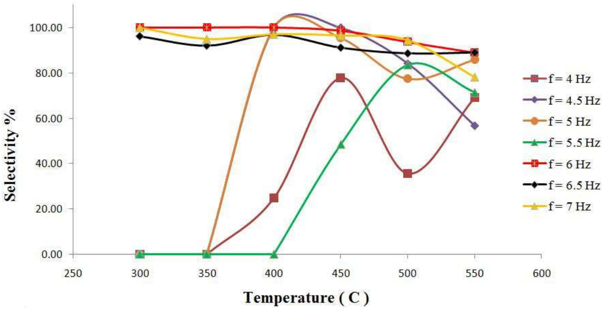

Fig. 4. Graphic selectivity (% weight) vs. temperature (^{0}C)

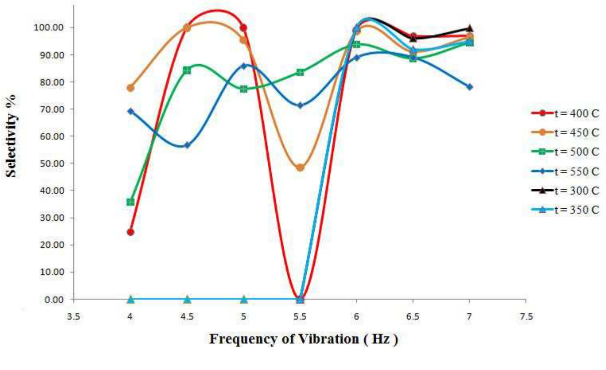

Fig. 5. Graphic selectivity (% weight) vs. frequency of vibration (Hz)

The selectivity (the amount of the produced gasoline and diesel fuel, divided by the amount of reacted gasoil) of light oilproducts was maximal at studied range of temperatures 300^{0}C, 350^{0}C, 400^{0}C, 450^{0}C, 500^{0}C, 550^{0}C and at a frequency MCA of 6 Hz. At 350^{0}C and a frequency of 6 (fig. 4) selectivity reached 99.99 %.

At range of frequency 4-5.5 Hz and a temperature 300 - 350°C , the selectivity was 0% , at a frequency 4 Hz, the selectivity reached 77.88% at 450°C, at a frequency of 4.5 and 5 Hz, the selectivity was at a range 95 - 99.95% at 400 - 450°C, respectively (fig. 5). At 5 Hz the selectivity decreased from 71% at 550°C to 48% at 450°C, at a frequency range 6 - 7 Hz the selectivity was at a range 78 - 99.99% where at 6 Hz and 350^{0}C was the higher value of selectivity.

Comparison of industrial process of catalytic cracking and AnCVB is shown in table. As seen from the table by using Si/Zr catalyst, reactor volume is reduced by 15 times in comparison with the heterogeneous catalysis and at 2.66 times in comparison with catalytic cracking at conditions of AnCVB on catalyst Nexus-345p and at 1.94 times in comparison with AnCVB on catalyst CaA zeolite.

Also on Si/Zr catalyst it was able to practically lower the process temperature, in comparison with the industrial indicators and at conditions of AnCVB on the catalysts: CaA zeolite and Nexus-345p.

Another important indicator is the high selectivity of process at conditions of AnCVB on Si/Zr catalyst of 98.7%wt, however due to the relatively low degree of conversion per one pass (67%wt) in the industrial conditions it requires a partial recirculation of raw material.

It should be noted that our studies (Si/Zr-catalyst, sample No:2) demonstrated improving the technological characteristics of cracking process in comparison with sample No:1. Despite a slight increase in temperature (the recommended value for the sample No: 2 is 350^{0}C), the new modification allows to conduct the process with a conversion degree of 76.4 (at sample No:1 the value of X was 67%) with preservation of selectivity higher than 99.5%). This will allow in the industrial conditions to carry out the process of catalytic cracking at aerosol nanocatalysis on a new modification of catalyst without using the recirculation stage of the raw material.

Table. The comparison of industrial process of catalytic cracking and AnCVB technology

Parameter	Technology					
	Industry	AnCVB				
Productivity ton/day	6300	6300				
1.Temperature, °C						
In reactor	640 - 525	550	300	600	630	350
In regenerator	640	not needed				
2. Catalyst	Nexus-345p	Y-Zeolite	Si/Zr No:1	CaA	Nexsus-345p	Si/Zr No:2
Concentration of catalyst in reactor, kg/m^3	300-700	$3 \cdot 10^{-3}$	$3 \cdot 10^{-3}$	$2 \cdot 10^{-3}$	$2 \cdot 10^{-3}$	$3 \cdot 10^{-3}$
3. Constant of cracking speed, s^{-1}	-	0,002	0,18	0,17	4,41	0,2
4. Selectivity of light products, % wt :	74,9	93,7	98,7	84,5	85,2	99,9
5. Degree of conversion, % wt :	with recycle	for one pass				
	85	15,7	67	95	89,1	76,4
6. Volume of reactor, m^3	800			306	420	158
regenerator, m^3	1600	Does not exist				

CONCLUSIONS

1. It was shown the possibility of obtaining light oilproducts using the technology AnCVB on a new modification of Si/Zr catalyst .

2. The perspective regime for designing an industrial unit working on Si/Zr catalyst, is a temperature 350°C, and a frequency of MCA 7 Hz at amplitude of MCA 12-14 mm, since in these conditions could reach the maximal degree of conversion of raw materials - 80%, and maintain a high output of light oilproducts - 76%wt.;

3. The organization of the catalytic cracking at technology of AnCVB on Si/Zr catalyst is promising at a number of parameters:

- It was shown that the process of catalytic cracking at conditions of AnCVB on Si/Zr, can be carried out at 300°C;

- The achieved selectivity of light oilproducts was more than 98%, due to the fact, that the gaseous phase is practically did not formed;

4. It was shown that applying of MCA at cracking process can intensify the speed of chemical reactions. So by using Si/Zr catalyst, it was possible to increase the degree of conversion of vacuum gasoil from 0 to 66.2% by increasing the vibration from 4 to 5.5 Hz at 300°C .

5. For gasoline fraction obtained on Si/Zr catalyst, the given indicators of octane numbers by research and motor methods were compliant with the standards of catalytic cracking gasoline.

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ПРИМЕНЕНИЕ АЭРОЗОЛЯ НАНОЧАСТИЧЕК SiO_2/ZrO_2-КАТАЛИЗАТОРА ДЛЯ ПОЛУЧЕНИЯ БЕНЗИНА И ДИЗЕЛЬНОГО ТОПЛИВА ИЗ ВАКУУМНОГО ГАЗОЙЛЯ

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Аннотация: Рассмотрено применение технологии аэрозольного нанокатализа для осуществления нового высокоэффективного процесса каталитического крекинга с получением бензина и дизельного топлива из вакуумного газойля. Температура в новом процессе ниже на 250^0C, чем в промышленных процессах крекинга, а количество катализатора снижается до концентрации 2,38 г/м^3 реактора. SiO_2/ZrO_2-катализато в условиях новой технологии показал селективность образования светлых продуктов свыше 99%.

Ключевые слова: крекинг, бензин, дизельное топливо, аэрозольный нанокатализ.