Comparing Degree of Conversion of Vacuum Gasoil on several Catalysts in AnC Technology

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Summary. The paper describes the experimental studies conducted using bi-metallic oxide SiO_2/ZrO_2-A-69-1 catalyst for catalytic cracking of vacuum gasoil using technology of aerosol nanocatalysis on vibrating bed reactor with conditions of temperature ranging from $300^{o}C$-$550^{o}C$ and mechanical chemical activation frequency ranging from 5.5-6Hz. Influence of temperature and variation in mechanical chemical activation frequency was observed in order to ascertain how such influences affects the degree of conversion of vacuum gasoil to light products. By controlling the oscillation frequency in the vibrating-bed reactor and regulating temperature, the activity of the bi-metallic oxide SiO_2/ZrO_2-A-69-1 catalyst increased, chemical reaction rate increased, which invariably increased the degree of conversion. Based on the experimental result in this work with bi-metallic oxide SiO_2/ZrO_2-A-69-1 catalyst, the highest degree of conversion to light products was obtained at temperature of $300^{o}C$ and at frequency of 5.5 Hz. On the basis of experimental studies and known facts about the peculiarities of the process the technological scheme has been developed for the pilot unit of the catalytic cracking of vacuum gas oil via aerosol nanocatalysis technology .

Key words: aerosol nanocatalysis, catalytic cracking, vacuum gasoil, bi-metallic oxide, mechanical-chemical activation

INTRODUCTION

Due to the growing demand for transportation fuels, such as gasoline and diesel, FCC (Fluidized Catalytic Cracking) continues to play a very major role in integrated refinery, as the primary conversion process of crude oil to lighter products. In the next 2 -3 decades, FCC process will likely be used for biofuels and possibly for reducing CO_2 emissions [1, 17, 19].

Catalytic cracking is the most important conversion facility in a modern refinery. The process consist of scission of the hydrocarbon C-C bonds present in the feedstock's (usually vacuum gas oil or residue) in order to obtain gasoline light alkanes or other low molecular weight hydrocarbons [2, 6, 8]. The feedstock fluid catalytic cracking (FCC) is often the gas oil portion of the crude oil that commonly boils within temperature range of 300 – $500^{o}C$. The 1st commercial FCC was acid treated natural clay, later synthetic silica alumina materials containing 10 – 15% alumina replaced the natural clay catalyst because synthetic silica alumina catalyst were shown to be more stable and yielded superior products [3, 9, 10]. In the

1950's, alumina–silica catalyst containing 25% alumina came into use because of their higher stability. These synthetic catalyst were amorphous, and their structure consisted of random array of silica and alumina in a tetrahedral coordination. Some minor improvements in yields were achieved by switching to catalyst such as magnesia– silica and alumina–zirconia–silica.

The breakthrough in FCC catalyst was the use of X and Y zeolites during the 1960's. The addition of the zeolites substantially increased catalytic activity and selectivity. In the modern units of catalytic cracking only zeolite containing aluminum-silicate catalysts with rare-earth metals are used [3]. However, the industrial organization of catalytic cracking has the following draw-backs:

(I) Necessity for a catalyst regenerator whose volume is twice that of the reactor volume (e.g. in order to process 2 million tons of raw materials per unit (G-47-103/MI), the volume of reactor must be $800 – 1600 m^3$;

(II) Necessity for a permanent catalyst supply since after a few seconds of activity there are, additional losses of the catalyst due to abrasion occur;

(III) The usage of high concentration of catalyst (about $700 kg/m^3$ of reactor);

(IV) The necessity of steam injection into the reaction stripping zone to ensure stripping of cracking products from the catalyst surface in the amount of 0.5 – 0.75 g per ton of raw materials.

Catalysts created from zirconium oxide was developed at the Institute for Sorption and Problems Endoecology Ukraine, and the reason for its development was to improve the yield of gasoline in industrial catalytic cracking of vacuum gas oil.

Various catalytic properties analysis were carried out whereby its investigation included , specific surface area of catalyst, pore size, ratio of $SrO_2 ._{.} ZrO_2$, methods of research on catalytic cracking process, and also research on the efficacy of aerosol nano catalysis technology AnC, and all these was done as to know the most effective and cheapest way in which catalytic cracking of vacuum gas oil can be conducted and also to produce a system in which high degree of conversion to light products can be achieved.

Continuous mechanical-chemical activation of catalyst surface in-situ which is a unique feature of the technology of AnC , leads to generation of reaction space of catalytically active nanoparticles ranging in size from 8 to 100nm [4, 5, 11, 12] , which can also increase the rate of chemical reactions to 10^6 times per mass of the catalyst.

ANALYSIS OF PUBLICATIONS
ON THE SUBJECT OF RESEARCH

The first experimental study conducted on the cracking of vacuum gas oil using technology of aerosol nanocatalysis, showed that even with the use of Al_2O_3 catalyst particle, which is a traditional heterogeneous catalyst usually not 100 % catalytically active for cracking hydrocarbon, but with variation in MCA frequency, it was shown to have a higher than usual degree of conversion to light products.

Further experiments found that the simple zeolites (CaA, NaX, type Y) when under conditions of aerosol nanocatalysis, which involves a constant mechanical-chemical activation on the surface of the catalyst by grinding of the surface of the catalyst with moving solid materials, shows an increase in the catalytic cracking reaction rate and reduction in temperature of the process, even in comparison with the industrialized fluidized catalytic cracking process on the modern multi-functional catalyst Nexus- 345p. Using the Nexus-345p (and its industrial components) in terms of AnC, observed was an increase in the output of light oil to 1.14 times and also increase in the reaction rate (per reactor volume) to almost 2.5 times. [5, 14-16].

The aim of this study was to compare the degree of conversion of vacuum gas oil with SiO_2/ZrO_2 A-69-1 bi-metallic oxide catalyst with previous catalyst WO_3/Zr-Si and Si/Zr used in previous study by using the parameters of temperature 300-550°C and MCA frequency 5.5-6Hz and to ascertain if variation in temperature and MCA frequency can result to high degree of conversion. And to find out if even at low temperatures of 300, 350°C with the 3 catalyst SiO_2/ZrO_2 A-69-1 bi-metallic oxide catalyst, WO_3/Zr-Si and Si/Zr there can be a degree of conversion.

It is important to note that the mechanical chemical activation of catalyst occur by forced vibration of the reactor, and that, in a vibrating bed reactor, there is the flow of reactants which is always approaching ideal mixing. In addition to that, aerosol nano-catalysis technology requires a small mass of metallic oxides nano-catalyst particles in small amounts of $0.3 – 10$ g/m^3 reactor instead of large volumes of existing industrial catalyst on supports with additives of noble metals. This will reduce the capital and operating costs, decrease the production cost, as well as, open up the possibility of excluding the catalyst regeneration and re-circulation stages from technical scheme of the process since catalysts constantly undergo continuous mechanical-chemical activation and display catalytic activity $10 – 100$ times higher than catalytic cracking on supports, because the produced coke will not be fixed on the active surface of the catalyst particles since the nanoparticles do not have pores. Also, aerosol nano-catalysis technology 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 thermodynamically possible [6, 18-21]. Conclusively, under the conditions of aerosol nano-catalysis technology, selectivity to light products is attainable even at a low temperature regardless of variations in the mechanical - chemical activation frequency depending on the catalyst used.

THE PURPOSE AND OBJECTIVES OF THE STUDY

The aim of study is to find the optimal process conditions for catalytic cracking new modification SiO_2/ZrO_2 - catalyst (laboratory sample A-69-1) using technology AnC, and to compare the degree of conversion of SiO_2/ZrO_2 - catalyst (laboratory sample A-69-1) with the degree of conversion of WO_3/Zr-Si and Si/Zr catalyst.

Research objectives:

- Determine at which temperature and frequency there is highest degree of conversion to light products using new laboratory sample (A-69-1) SiO_2/ZrO_2 – catalyst.

- Determine if through variation in MCA frequency there can be catalytic cracking of vacuum gas oil at low temperatures like 300°C, 350°C using new modification SiO_2/ZrO_2 - catalyst (laboratory sample A-69-1).

- Compare the degree of conversion of SiO_2/ZrO_2-A-69-1 with Si/Zr and WO_3/Zr-Si at MCA frequencies of 5.5Hz and 6Hz, since such frequencies favor highest degree of conversion.

THE METHODOLOGY OF EXPERIMENTAL
RESEARCH AND ANALYTICAL CONTROL
OF THE REACTIONS PRODUCTS

Laboratory installation process for cracking vacuum gas oil under aerosol nano catalysis technology is no different from other laboratory set-up of catalytic cracking, which consist of a reaction unit for dosing of raw materials and a receiving unit for collection of reaction products [7]

The peculiarity of the experimental work is preliminary preparation of the catalyst system. Experimentally the catalytically active material is adsorbed on the surface of the dispersing material in reactor [7] before the experiment is conducted, "Pollination" which is the adhesion of the catalytically active material to the surface of the dispersing material, so that the surface can achieve saturation. Catalytic active component sample is added to the dispersing material. Pollination is performed at operating temperatures and mechanical chemical activation frequency, until the weight of the catalytically active material does not change with each unloading of the catalytic system reactor after separation from dispersing material.

Mass of the catalytic sample if constant after unloading, indicates that there was a complete saturation of the surface of the dispersing material and as a result, all catalytic active component had been subjected to mechanical-chemical activation and this is the science behind technology of aerosol nano catalysis. This indicates a regeneration of catalyst has occurred "in situ".

Petroleum products obtained from [7] were distilled by distillation apparatus for ULAB-1-42A [8].

Defined output gasoline fraction begins from (temperature 80°C-180°C) and diesel fractions (temperature 180°C-350°C) and heavy residue is (temp >350°C). For analytical determination of gaseous reaction products chromatography LHM.8 500-color (ethylene and hydrogen were determined with an accuracy of 0.01% vol.) the composition of gasoline fraction (research method) was determined in chromatograph "CRYSTAL 5000".

Mass of liquid petroleum is determined after measuring the volume fraction and density of its pycnometric:

$$m_{df} = \rho_{df}.v_{tvf} , \qquad (1)$$

where: ρ_{df} = Density fraction, (g/ml)

V_{tvf}= total volume fraction, (ml)

The volume of gaseous fraction is determined after determining the volume fraction of each component in the gas mixture chromatography method:

$$V_{gf} = X_{scgp} \cdot V_{tvgf} , \qquad (2)$$

where: X_{scgp}= substance of content in gas phase (% volume)

V_{tvgf}= total volume of the gas fraction (liter)

Mass of coke was determined by weighing the catalyst system after the experiment using the formula:

$$M_{cokes} = M_2 - M_1, \qquad (3)$$

where: M_2 = mass of the catalyst system after the experiment (gm.)

M_1=weight of the catalyst system before experiment (gm.)

The mass output of each hydrocarbon fraction determined by the formula:

$$X = \frac{M_{fraction}}{M_{weight}} \times 100\% , \qquad (4)$$

where: $M_{fraction}$ = mass of fraction formed as a result of the experiment (gm.)

M_{weight} = weight of vacuum gas oil, which was used for experiment (gm.)

The selectivity to light oil was determined by:

$$\phi = \frac{M_{gasoline+diesel\,fractions}}{M_{trm}} , \qquad (5)$$

where: $M_{gasoline+diesel\,fractions}$ = mass of gasoline and diesel fractions respectively (gm.)

M_{trm} = transformed raw mass (the mass of the reaction products – the exception of heavy residue, gm.)

To determine the kinetics of cracking petroleum fractions :

$$W = K \cdot C_{CCR} = K_0 \cdot C_{CCR} \cdot e^{\frac{-E}{RT}} , \qquad (6)$$

where: C_{ccr} = conventional concentration of raw materials in the reactor,

K= rate constant for the formation of this component,

K_0 = coefficient of frequency

With the inclusion of the kinetic equation, parameters of aerosol technology nanocatalysis formula takes the form:

$$W_{scgp} = K \cdot C_{ccr} \cdot K_v = K_0 \, K_v \cdot C_{ccr} \cdot e^{\frac{-E}{RT}} \qquad (7)$$

where: K_v = parameter of vibration, depending on the frequency (f) amplitude (A) and others in the form of $K_v = K*f^{\,n} * A^{\,m}$

After determining experimentally the rate of substance or fractions and knowing the concentration of material in the reactor, we can determine the rate constant of the reaction. This will allow future industrial reactor calculate the contact time for a given degree of conversion of raw materials. Contact time is a parameter for evaluating aerosol nanocatalysis reactor dimensions and for comparison with industrial technology.

Productivity - the amount of substance produced per unit of time - can be attributed to the weight or volume of the catalyst reactor:

$$P_1 = \frac{M_{substance\circ content\triangleright in\circ gas\triangleright phase}}{t \cdot V_{r-rn}} \qquad (8)$$

where: M = mass of substance / fractions (kg.),

Vr-rn = volume of reactor (m^3),

t= duration of the experiment (hours).

$$P_2 = \frac{M_{substance\circ content\triangleright in\circ gas\triangleright phase}}{M_{cat}} \qquad (9)$$

where: M_{cat} = mass of catalyst in reactor (kg.)

Specific performance makes it possible to compare technologies and equipment.

EXPERIMENTAL PART

The pre-heated vacuum gas oil was fed into the reactor (5) by a syringe-batcher (1), which is located in the thermal cabinet (2), and it was then subjected to thermal treatment by heater (13). Cracking reactions took place inside the reactor. Experiments were conducted at temperatures: 300°C, 350°C, 400°C, 450°C, 500°C, 550°C, and at frequencies between 5.5 – 6.0 Hz. The temperature in the reaction zone was measured by a thermocouple (11), and sustained by a regulator (12). In order to keep the catalyst inside the reactor, a metal-cloth filter (6) was placed at the bottom of reactor, the liquid reaction products pass through a water-cooled condenser

(7) and were then gathered in a flask for the liquid fraction (8). The reaction products were two fractions – liquid and gas. The non-condensed gases from the cracking process go through the sampling point (9), and were then sent to the gas washer (10) where they bubble through the water layer. The gases were then directed into a gas container (15), and passed through container with water (14). The reactor moves upward and downward in a back-and-forth linear motion with the help of vibrating device (4). The oscillation frequency and temperature were set and controlled by a regulator (12). The liquid phase wassubjected to fractional distillation so as to separate the resulting mixture into their individual component by using

an apparatus ULAB 1–42AThe content of the gaseous products of the reaction were analytically determined by using calibrated gas chromatographs such as LHM–8, COLOR–500. Ethylene (C_2H_6) and hydrogen (H_2) were determined with an accuracy not less than 0.01 *vol.* %. The products composition and octane number of the gasoline fraction (*research and motor methods*) were determined by chromatograph CRYSTAL–5000.2.

The catalytic cracking of vacuum gas oil under conditions of aerosol nano catalysis technology was performed at temperatures between $300 – 550^oC$ using the equipment above in Fig 1, mechanical-chemical activation (MCA) frequency within 5.5 to 6.0 Hz over bi-functional SiO_2/ZrO_2 catalyst in a vibrating bed reactor, but for this study, MCA frequency 5.5 and 6Hz were taken into consideration, because it was observed at this frequency all 3 catalyst showed a considerable and significant degree of conversion between each other. The results of the experiment are presented in the graph below.
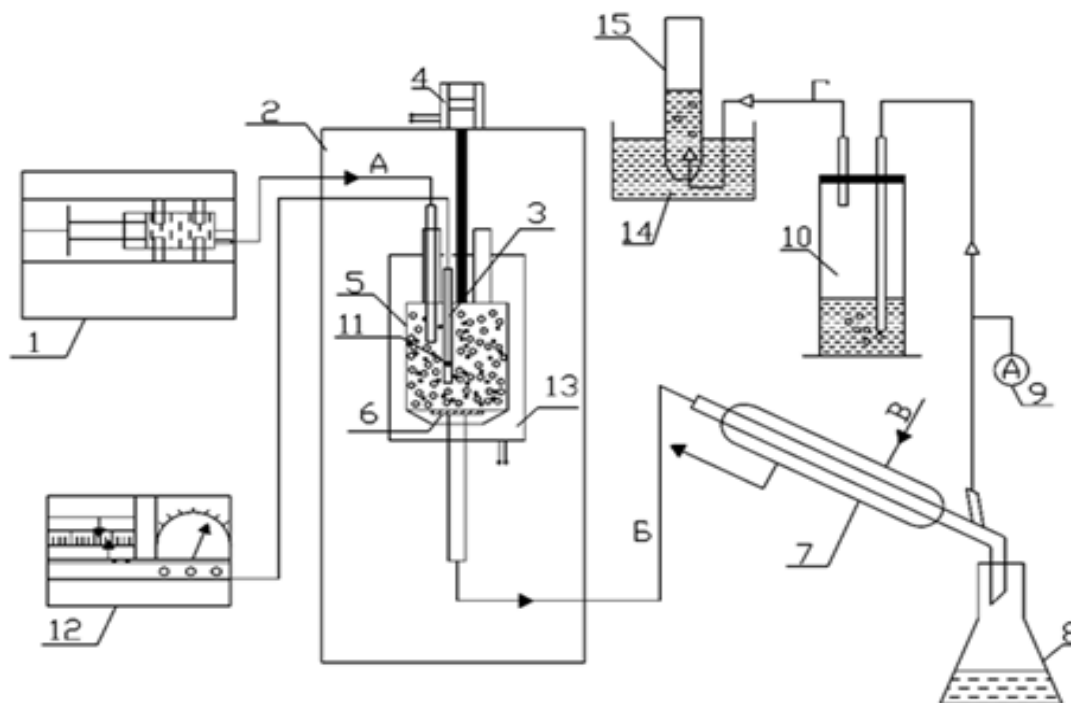

Fig 1. Schematic diagram of the pilot unit
1 – Syringe batcher, 2 – Thermal cabinet, 3 – Pocket of thermocouple, 4 – Vibrating device, 5 – Reactor, 6 – Metal-cloth filter, 7 – Water condenser, 8 – Receiver of liquid fraction, 9 – Sampling point, 10 – Gas washer, 11 – Thermocouple, 12 – Regulator for oscillation frequency and temperature, 13 – Heater, 14 – Water container, 15 – Gas receiver
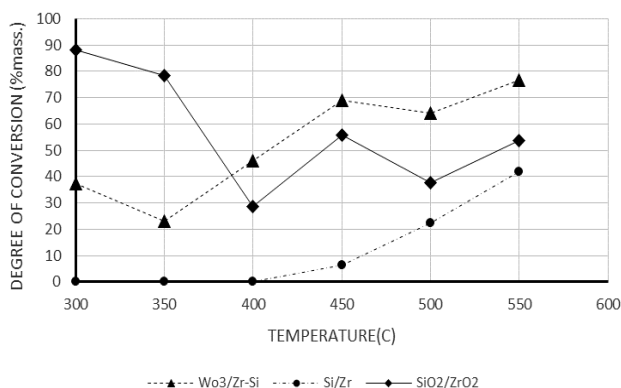

Fig. 2. Degree of conversion on MCA frequency (5.5Hz) vs. temperature using SiO_2/ZrO_2 A-69-1 and comparing with Si/Zr and WO_3/Zr-Si

The maximum % mass degree of conversion here is observed at 89% mass with catalyst SiO_2/ZrO_2 A-69-1 and at a temperature of 300^0C, and this in contrast with the other catalyst samples in which the highest degree of conversion was observed at 41% (550^oC) Si/Zr and 79% (550^oC) WO_3/Zr-Si respectively. On this graph, it can also be noted that Si/Zr catalyst did not show any degree of conversion at 300^oC, 350^oC, 400^oC and also that after 400^oC, there was a gradual increase in degree of conversion from 450^oC to 550^oC, but with WO_3/Zr-Si catalyst there was irregularities in its degree of conversion.
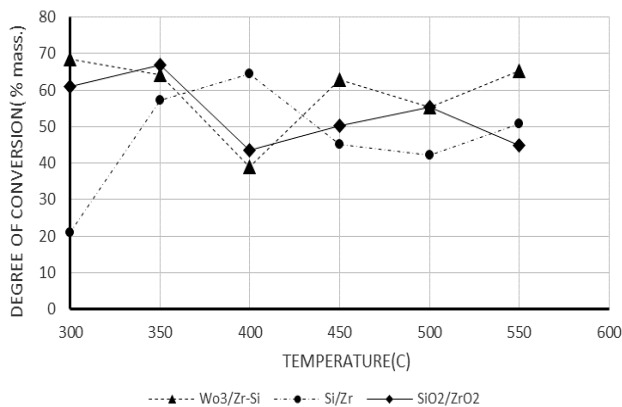
Fig. 3. Degree of conversion on MCA frequency (6Hz) vs. temperature using SiO_2/ZrO_2 A-69-1 and comparing with Si/Zr and WO_3/Zr-Si

The maximum degree of conversion of 70% was observed at 300^0C but with catalyst sample WO_3/Zr-Si in comparison with SiO_2/ZrO_2 A-69-1 catalyst sample in which the highest degree of conversion was at 350^0C at was recorded at 68% , while for Si/Zr catalyst the highest degree of conversion was recorded at 400^0C and 66%. On this chart it can be seen that WO_3/Zr-Si gave the highest degree of % mass conversion, and its % mass conversion never went below 40%, while with Si/Zr the lowest % mass conversion was at 21% at 300^0C.

From the results in this study , it can be seen that SiO_2/ZrO_2 A-69-1 catalyst sample showed the highest mass degree of conversion of 89% at 300^0C at 5.5Hz,while WO_3/Zr-Si catalyst sample showed the highest mass degree of conversion of 70% at 300^0C at 6Hz, as this can be attributed to the fact that at low temperatures there is less intense thermal softening of the catalyst and as such there are more available sites for the reactants to react with on the surface of the catalyst, and also considering a gradientless reactor was used to conduct experiment, it can be said, this enabled the reaction to approach ideal mixing , and also that even at MCA frequency of 6Hz, SiO_2/ZrO_2 A-69-1 catalyst sample still showed a significant degree of% mass conversion of 68%, that goes to say that the SiO_2/ZrO_2 A-69-1 catalyst has a great affinity to % mass degree of conversion to light products at low temperatures. While Si/Zr catalyst showed low % conversion(at 6Hz) or no % mass conversion (at 5.5Hz) of vacuum gasoil to light products at low temperature, that is to say the catalyst activity cannot be relied upon at low temperatures in comparison with the other 2 catalyst, maybe as a result of the reactors previously used.

On the basis of the known facts about the technology of aerosol nano catalysis and based on our own research was developed technological scheme of the pilot plant for catalytic cracking of vacuum gasoil (Fig. 4).

This scheme will consist of: **E1**=Bunker for gas oil, which is where the feed pre heat will take place, **E2**=Bunker for storage of catalyst, **H1**=Charge pump for supply of gas oil, **H2**=Dispatcher for catalyst, **R**=Reactor aerosol nanocatalysis AnC, **F**=Filter that will serve to separate the catalyst from the cracked hydrocarbon vapors. **H3**=Pump around, **K1**=Rectification column,

K2=Regenerator, **H4**=Compressor for the supply of air, for the regeneration of catalyst, and this will be done at full combustion, with

the excess reaction component being oxygen. **C1**=Cyclones, that provides the separation of the catalyst so as to be sent back to **E2**. **T**=Tube, **T1**=Heater for heating of feedstock, so as to keep it always in fluid state, **T2**=Additional heater.

1=Gas, **2**=Air, **3**=Gasolineproducts,**4**=Diesel products, **5**=Heavy gas oil, **6**=Heated water, **7**=Technical water.
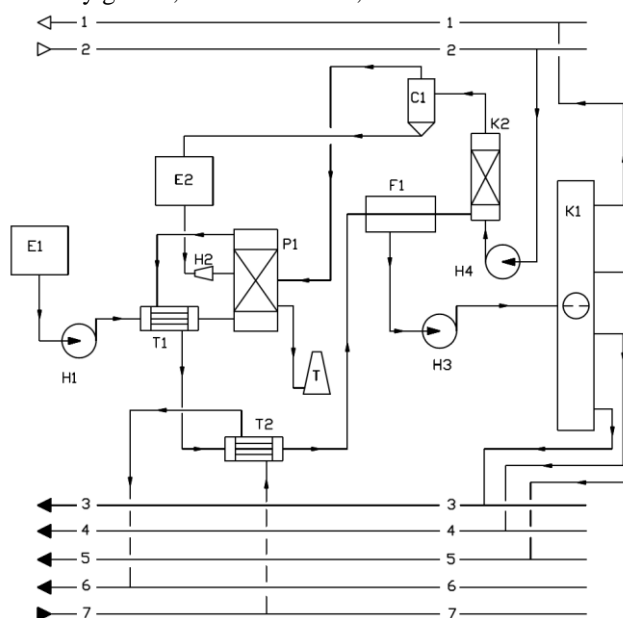

Fig 4. Proposed technological scheme of industrial catalytic cracking unit of vacuum gas oil using technology of AnC

The main difference between this technology from traditional industrial processes of cracking is the small size of the regenerator, and difference is the implementation of the mechanical activation of the catalyst in a reactor.

CONCLUSIONS

1. Degree of conversion of vacuum gasoil to light products is possible at low temperatures of $300^oC,350^oC$ using catalyst: SiO_2/ZrO_2 A-69-1 and WO_3/Zr-Si even with variation in MCA frequency from 5.5-6Hz.

2. SiO_2/ZrO_2 A-69-1 catalyst showed the highest % mass degree of conversion of 89% at 300^oC at 5.5Hz, while WO_3/Zr-Si catalyst sample showed the highest % mass degree of conversion of 70% at 300^0C at 6Hz.

3. 3) It can be seen in comparison of SiO_2/ZrO_2 A-69-1 catalyst with WO_3/Zr-Si catalyst and Si/Zr catalyst that SiO_2/ZrO_2 A-69-1 catalyst showed a greater advantage above the other 2, in that % high mass degree of conversion is guaranteed at low temperatures and variation in frequency.

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СРАВНЕНИЕ СТЕПЕНИ ПРЕВРАЩЕНИЯ ВАКУУМНОГО ГАЗОЙЛЯ ДЛЯ РАЗЛИЧНЫХ КАТАЛИЗАТОРОВ В УСЛОВИЯХ ТЕХНОЛОГИИ ANC (АЭРОЗОЛЬНЫЙ НАНОКАТАЛИЗ)

Филипс Тобенна Химдиади, С. Кудрявцев

Аннотация. В статье изложены результаты экспериментальных исследований, проведенные с применением биметаллического оксидного катализатора SiO2/ZrO2 (лабораторный образецA-69-1), применительно к процессу каталитического крекинга вакуумного газойля в условиях технологии аэрозольного нанокатализа с реактором виброожиженного слоя. Условия экспериментальных исследований: температура 300oC-550oC; интенсивность механохимической активации катализатора 5,5-6 Гц. Эксперименты проведены с целью выявления влияния температуры и интенсивности механохимической активации на степень конверсии вакуумного газойля в светлые продукты. Управляя частотой колебаний вибрреактора и регулируя температуру удалось увеличить активность катализатора SiO2/ZrO2(лабораторный образецA-69-1) и соответственно повысить степень конверсии. Наивысшая степень конверсии для исследованного образца в условиях эксперимента была достигнута при 3000С и частоте механохимической активации 5,5 Гц. На основе экспериментальных исследований и известных фактов об особенностях процесса крекинга

в условиях аэрозольного нанокатализа на других катализаторах была разработана технологическая схема для опытной установки каталитического крекинга вакуумного газойля с использованием технологии аэрозольного нанокатализа.

Ключевые слова: аэрозольный нанокатализ, каталитический крекинг вакуумного газойля, биметаллические оксиды, механохимическая активация.