An FCC Catalyst for Maximizing Gasoline Yield

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‖ Abstract

A Y zeolite-containing wide-pore composite was synthesized by *in situ* technique using polyvinyl alcohol and silica sol material. A fluid catalytic cracking (FCC) catalyst for maximizing gasoline yield was successfully developed with the modified composition. The as-made zeolite Y in its sodium form had a relative crystallinity of 52.9 % with a high silica/alumina amount of substance ratio of 5.7. The FTIR analysis showed that the acid distribution of the catalyst modified with rare earth, phosphorus and steaming stabilization process was more concentrated on the range of intermediate and strong acidity. This kind of modification can direct more hydrocarbons to enter into the pores to be converted, as well as remarkably increases the possibility of gasoline formation through a cracking reaction. The nitrogen adsorption–desorption analysis showed that the catalyst had more meso- and macro-pores distribution, which can effectively reduce the mass-transfer resistance in the reaction process and accelerate the diffusion of the product molecules. The evaluated results indicated that the prepared catalyst could decrease the excessive cracking of middle distillate and improve the gasoline yield effectively. The gasoline yield by mass had increased by 2.69 %, while the coke yield and dry gas yield had decreased by 0.91 % and 0.19 %, respectively, and then the olefin content by volume in the cracked gasoline reduced by 4.6 %, the research octane number (RON) and motor octane number (MON) increased by 0.6 and 0.5, respectively. The good product selectivity and higher gasoline yields of the prepared catalyst were obviously related to its wide pore structure and its optimized acidity distribution.

‖ Keywords

FCC, catalyst, gasoline, modification

1 Introduction

The product selectivity of fluid catalytic cracking (FCC) units is one of the main topics to be understood and controlled in order to increase process benefits and comply with mandatory regulations on the composition of transportation fuels.[1–3] China is one of the fastest developing countries in the global petrochemical industry. The refining capacity ranked second place. Although refining capacities continue to grow, the domestic fuel oil demands are not being met and a large number of high quality refined oil still needs to be imported. One of the reasons for this is the rapid development of the automobile industry in recent years. In the next decade, it is expected that gasoline from FCC units will continuously increase. Therefore, both process changes and new catalysts need to be considered together to improve the gasoline yield of FCC processes. It is urgent to develop a novel FCC catalyst for producing higher gasoline yield.

The modern FCC catalyst consists, in general, of two major components: zeolite and matrix. The zeolite used in FCC catalysts are mostly synthetic, faujasite-type zeolite Y and high silica Y zeolites, which is the major contributor to the catalytic activity and selectivity of the FCC catalyst. The activity and selectivity of the FCC catalyst come from the acid sites and the pore structure, respectively. Acid sites of zeolite Y can be generated in the framework, and their strength and concentration can be tailored for a particular application. Their intricate channel structure allows the zeolite to present different types of shape selectivity, *i.e.*, product, reactant, and transition state, which can be used to direct a given catalytic reaction toward the desired product avoiding undesired side reactions. The acidic properties and porous properties of zeolite have always been the subject of numerous studies.[4–10]

Catalytic cracking is an acid-catalysed reaction, which means that the density of the acidic active sites and their locations will affect the rates of all catalytic processes and reaction pathways. The loading, position, occupation, and coordination of trivalent rare earth cation on Y zeolite directly affect not only the acid amount (density of acid centres), acidity, and the ratio of Brønsted acid to Lewis acid, but also the catalytic activity of the FCC catalyst. The acid distribution of P-RE-USY is more concentrated in the range of intermediate and strong acidity, improving the hydrogen transfer activity. The reduction of strong acid decreases the formation of coke.

The aim of the current work was to study the preparation and modification of catalyst, and to investigate the catalytic properties. The acid strength and pore distribution of FCC

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catalyst for maximizing gasoline yield were investigated. The acid distribution of catalyst was more concentrated on the range of intermediate and strong acidity. The modified catalyst possessing wide-pore distribution could effectively reduce the mass-transfer resistance in the reaction process and accelerate the diffusion of the product molecules.

2 Experimental

2.1 Materials

Kaolin was obtained from China Kaolin Co. Ltd. Polyvinyl alcohol above 2000 degrees of polymerization and diammonium hydrogen phosphate were obtained from Sinopharm Chemical Reagent Beijing Co. Ltd. Silica sol was obtained from Changsha Xingye Industrial and Trading Co. Ltd. Sodium silicate (containing 21.4 % SiO_2, 7.1 % Na_2O), sodium metaaluminate (containing 20.4 % Na_2O, 3.0 % Al_2O_3) and lanthanum chloride solution (containing 185 g L^{-1} $LaCl_3$) were obtained from the Yueyang Jucheng Chemical Co. Ltd.

2.2 Catalyst preparation

The catalyst preparation involved two steps: synthesis of NaY zeolite and its modification.

The NaY zeolite was prepared by *in situ* synthesis method. Kaolin, sodium silicate, silica sol, and polyvinyl alcohol were mixed thoroughly. The mixture was sprayed, acquiring microspheres whose average particles were about 60–70 μm in diameter. The microspheres were calcined at 930 °C for 2 h and 750 °C for 2 h, respectively. Next, the calcined microspheres were mixed together with sodium silicate, sodium hydroxide, zeolite initiator, distilled water, and the mixture was heated at 90–95 °C for 20–24 h to synthesize the NaY zeolite, then the zeolite was washed, filtered, and dried.

The synthesized zeolite was exchanged several times with ammonium chloride of mass fraction of approximately 10 to 30 to replace the sodium, as described elsewhere.[4] The sample was then exchanged with an aqueous solution of lanthanum chloride for 60 min, then an amount of diammonium hydrogen phosphate was added into the reaction mixture, and the mixture continued to react for 30 min. The mixture was filtered, washed, and calcined at 600 °C with 100 % steam for 2 h.

2.3 Characterization

1) X-ray diffraction: The relative crystallinity, silica/alumina, unit cell parameter of zeolite Y were recorded on a Rigaku Ultimi IV diffractometer with Cu radiation and a Ni filter. The relative crystallinity was determined using the ratio of diffraction peak intensity in the area of Miller indices {533}.

2) Nitrogen adsorption–desorption methods: The specific surface areas, pore volumes, and pore size distributions were measured on an ASAP 2020 sorptometer using adsorption and desorption isotherm plots at −196 °C. Prior to the measurement, the samples were degassed at 350 °C for 12 h. The surface areas were calculated using the Brunauer–Emmett–Teller (BET) method. The micropore volumes and external surfaces areas were calculated using the t-plot method. The pore parameters, mesopore surface and mesopore volume were calculated from the desorption branches of these isotherms using the BJH method.

3) FTIR test: The Brønsted and Lewis acid sites in the catalyst were recorded on an AVATAR 370 FT-IR spectrometer. The FTIR spectrometer was equipped with an *in situ* cell equipped with KBr windows. This article defines that the total acid amount and strong acid amount, respectively, is absorbance above 200 °C and 400 °C. The bands located at 1540 and 1450 cm^{-1} are used to estimate the amount of pyridine adsorbed on Brønsted and Lewis acid sites, respectively.

4) SEM: The morphology was determined by scanning electron microscopy (JEOL JSM-6360) after coating with an Au evaporated film.

2.4 Catalytic properties evaluation

The catalytic performance was tested on an advanced catalyst evaluation bench unit (ACE, Kayser Corp.) with a mixture of 70 % vacuum gas oil (VGO) and 30 % vacuum tower bottom (VTB) as the feed.[11] The catalyst was initially steam-deactivated at 800 °C for 17 h with 100 % steam. The reaction conditions: reactor temperature of 520 °C, WHSV of 19 h^{-1} and catalyst-to-oil mass ratio of 7.0.

3 Results and discussion

3.1 XRD

As shown in Fig. 1, the XRD patterns showed that the synthesized sample was faujasite-type zeolite Y. It can also be seen that the prepared catalyst and the reference catalyst exhibited the crystalline features of the Y. Other zeolite phases were not detected. In comparison with the XRD patterns of the reference catalysts, the prepared catalyst exhibited higher intensity peaks. In the present work, by using the unique combination of silica sol and kaolin, and *in situ* crystallization technology, a much higher Y-type zeolite content was obtained, the zeolite Y in its sodium form had a relative crystallinity of 52.9 % with a silica/alumina amount ratio of 5.7, the BET surface area and total pore volume were 558 $m^2 g^{-1}$ and 0.41 $cm^3 g^{-1}$, respectively. The silica alumina ratio of the zeolite relate to its catalytic properties intimately. With the silica alumina ratio of zeolite increasing, the surface acidity density decreased, the acid strength increased, and the frame stability of zeolite improved. Participation of silica sol is the contributor to higher silica/alumina amount ratio, and this would help to improve the attrition index of the prepared catalyst. The attrition index of the prepared catalyst was much better than that of the reference catalyst (Table 1). Good wear-resistance performance can improve the fluidization behav-

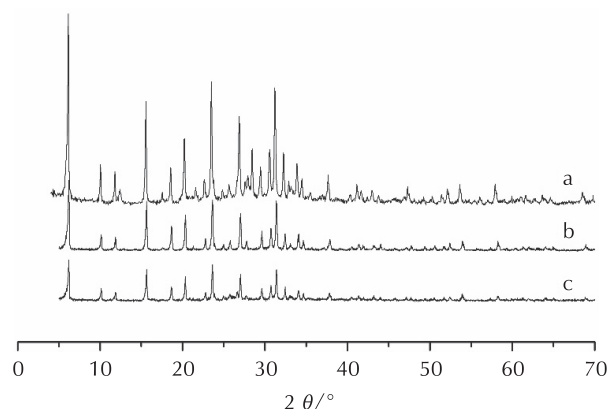iour and lead to less replenishment of fresh catalyst during the FCC process.

Fig. 1 – XRD patterns: a) synthesized NaY, b) prepared catalyst, and c) reference catalyst

Slika 1 – Rendgenski difraktogrami: a) sintetizirani NaY, b) sinteti- zirani katalizator, c) referentni katalizator

We chose a comparable commercial resid catalyst for max- imizing gasoline yield as reference catalyst. The physico- chemical characteristics of the two kinds of catalysts are listed in Table 1. The physical properties of the prepared catalyst were much better than those of the reference cat-

alyst, in comparison with the reference catalyst, the BET surface area and micropore surface area of the prepared catalyst had increased by 38.2 % and 60.0 %, respectively, total pore volume, micropore pore volume, and BJH pore volume had increased by 60.9 %, 80.0 %, and 85.7 %, respectively, and the prepared catalyst had larger average pore diameter. These advantages contribute to the good performance of the catalyst in the FCC process.

3.2 FTIR spectroscopy

The FTIR spectra of the catalysts are shown in Fig. 2. The spectra of the prepared catalyst had one broad band at about 460 cm^{-1}, representing the deformational vibration of Si–O bonds, and another band at about 580 cm^{-1}, typ- ically ascribed to the double six-member rings in micropo- rous zeolite Y,[12] suggesting the presence of primary and secondary structural building units of zeolite Y. The spectra of the reference catalyst had two bands at about 475 cm^{-1} and 545 cm^{-1}. Compared to the prepared catalyst, the spectral band appeared to shift. This difference inferred that the two materials and modification method may have different local structural environments in the primary and secondary structural building units.[13–14]
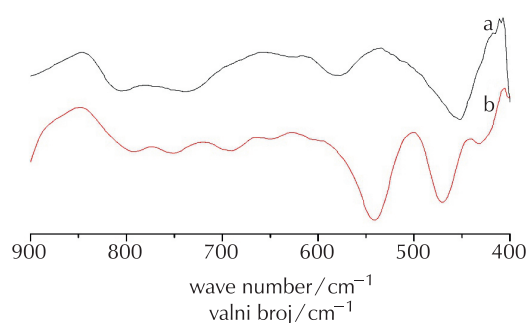

Fig. 2 – FTIR spectra of (a) prepared catalyst, and (b) reference catalyst

Slika 2 – Infracrveni spektri sintetiziranog (a) i referentnog (b) ka- talizatora

IR spectroscopy of adsorbed pyridine is a technique used for measuring and distinguishing different types of acid sites on catalyst surfaces. Table 2 lists the acid strength distributions of catalysts. In comparison with the refer- ence catalyst, the weak Lewis acid and total Brønsted acid amounts in the catalyst were higher, and the catalyst can decrease the strong Lewis acid amount. Strong Lewis acids induce undesirable coke production, but weak Lewis acid sites and higher Brønsted acid sites are needed for cracking selectivity. The good acidity of the catalyst has been attrib- uted to the modification by rare earth and phosphorus. After modification by La^{3+} and phosphorus, the strength of strong Lewis acidity in the catalyst became weaker. When the phosphorus reacted with zeolite Y, the hydroxyl con- nected with aluminum atoms on the zeolite surfaces was replaced by P–OH, and the acid strength weakened as the phosphorus content increased, because the acid strength of P–OH is weaker than that of Al–OH.[15]

Table 1 – Composition and properties of reference and pre- pared catalysts

Tablica 1 – Sastav i svojstva referentnog i sintetiziranog kataliza- tora

	Ref. catalyst Ref. katalizator	Prep. catalyst Sint. katalizator
chemical composition		
$w(Na_2O)/\%$	0.12	0.21
$w(Al_2O_3)/\%$	49.8	33.1
$w(RE_2O_3)/\%$	2.7	2.4
$w(P)/\%$	0.8	1.0
properties		
BET surface area/$m^2 g^{-1}$ površina po BET-u/$m^2 g^{-1}$	251	347
micropore surface area/$m^2 g^{-1}$ površina mikropora/$m^2 g^{-1}$	160	256
total pore volume/$cm^3 g^{-1}$ ukupan obujam pora/$cm^3 g^{-1}$	0.23	0.37
micropore volume/$cm^3 g^{-1}$ obujam mikropora/$cm^3 g^{-1}$	0.10	0.18
BJH pore volume/$cm^3 g^{-1}$ obujam pora po BJH-u/$cm^3 g^{-1}$	0.14	0.26
average pore diameter/nm prosječni promjer pora/nm	4.5	7.4
crystalline unit cell size/nm veličina jedinične ćelije/nm	2.455	2.452
attrition index/% atricijski indeks/%	1.6	0.7

Table 2 – Acidic properties of catalysts (relative strength of absorbance bands)
Tablica 2 – Kiselinska svojstva katalizatora (relativna jakost apsorpcijskih signala)

	Prepared catalyst Sintetizirani katalizator		Reference catalyst Referentni katalizator	
	Brønsted	Lewis	Brønsted	Lewis
weak acid amount slabo kisela aktivna mjesta	7.1	4.3	6.9	7.2
strong acid amount jako kisela aktivna mjesta	7.6	7.8	4.7	6.8
total acid amount ukupna kisela aktivna mjesta	14.7	12.1	11.6	14.0

3.3 SEM

Fig. 3 displays the SEM images of catalysts. It may be seen that the sizes of the Y zeolite particles are 0.2–0.7 microns, indicating that smaller crystals were evenly and densely packed on the surface of the kaolin microspheres, and agglomerated with the larger particles. The octahedral morphology of the Y zeolite was observed in all of the images. The SEM micrograph of the reference catalyst showed that the NaY zeolite had dispersed onto the compacted kaolin matrix, and some agglomerated with the larger particles. The NaY crystals content was less, and unevenly distributed.
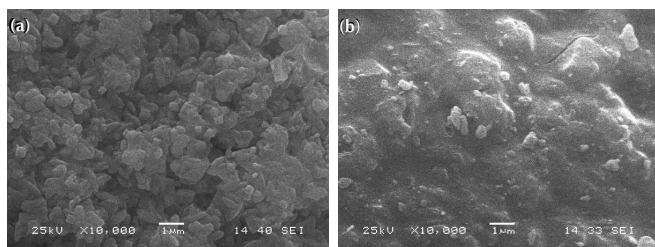

Fig. 3 – SEM images: (a) prepared catalyst, and (b) reference catalyst
Slika 3 – Elektronska mikrografija sintetiziranog (a) i referentnog (b) katalizatora

3.4 Pore structure

One of the most important properties for FCC catalyst is its pore characteristics. Fig. 4 showed the nitrogen adsorption–desorption isotherms of catalysts. The isotherm of the prepared catalyst exhibited the representative characteristics of type IV adsorption–desorption. The hysteresis loop that occurred in a relative pressure range of 0.50–1.0 was due to the presence of mesopores. The isotherms of the prepared catalyst exhibited a steeper decrease and a larger hysteresis loop than the reference catalyst, indicating that the prepared catalyst was richer in mesopores. The results confirmed that the residue of the caustic leached kaolin matrix and polyvinyl alcohol could endow the catalyst with more mesopores, and effectively improve the catalytic ca-

pability and stability of the catalyst.[16]

Fig. 5 shows the broad distribution of catalysts. The prepared catalyst possessed a hierarchical porous distribution, the distribution was concentrated on approximately 3.8, 5.6, 7.9–20 nm, respectively. This was mainly due to the addition of silica sol and polyethylene glycol during the synthesis process. The distribution of the reference catalyst was observed at approximately 4.0 nm. The presence of the mesopores in the crystallites of the catalyst should basically increase the accessibility of large molecules to the external opening of the pores. In other words, and from the standpoint of large reactant molecules, the presence of mesopores in the crystallites of the catalyst would be equivalent to increasing the external surface of the catalyst, making a larger number of pore openings accessible to the reactant. The unique pore system of the prepared catalyst should greatly enhance the accessibility of the catalytically active sites inside the microporous channels to larger reactant molecules, leading to the accelerated diffusion of products and fewer secondary reactions that lead to the formation of dry gas and coke.[17]
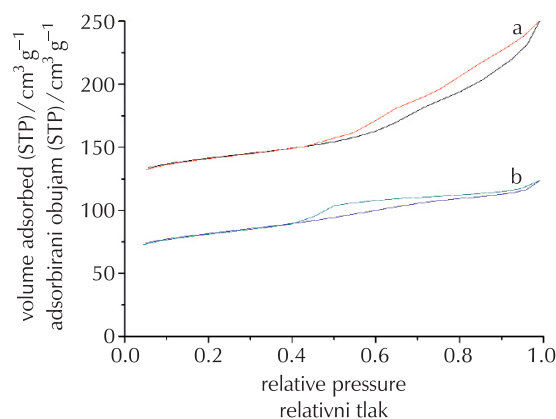

Fig. 4 – Nitrogen adsorption–desorption isotherms: (a) prepared catalyst, and (b) reference catalyst
Slika 4 – Apsorpcijsko-desorpcijske izoterme dušika za sintetizirani (a) i referentni (b) katalizator
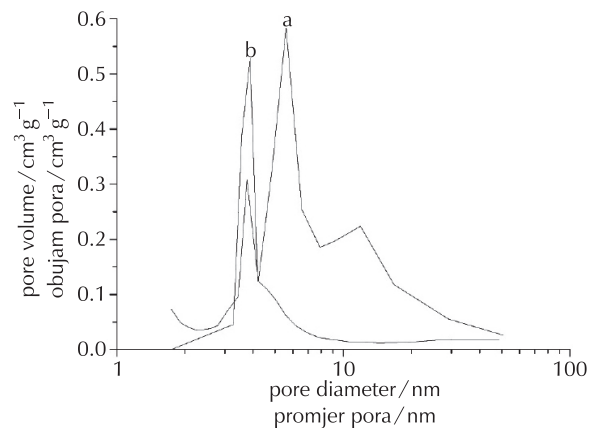

Fig. 5 – Pore size distribution: (a) prepared catalyst, and (b) reference catalyst
Slika 5 – Distribucija veličine pora sintetiziranog (a) i referentnog (b) katalizatora

3.5 Catalytic properties evaluation

The catalytic cracking results are shown in Table 3. In comparison with the reference catalyst, the prepared catalyst had a much lower HCO yield, the HCO yield decreased by 1.25 %, the gasoline yield was 2.69 % higher , while the coke yield was 0.91 % lower. The results indicated that the catalyst had better heavy oil cracking capability, and coke and dry gas selectivity. The good product selectivity and higher gasoline yields of the prepared catalyst were obviously related to its wide pore structure and its optimized acidity distribution. The properties of the cracked gasoline are summarized in Table 4. Comparably, the olefin volume in the cracked gasoline had reduced by 4.6 %, while the research octane number (RON) and motor octane number (MON) increased by 0.6 and 0.5, respectively. The above results showed that the prepared catalyst, containing the newly modified zeolite Y, had outstanding performance in reducing naphtha olefin content, as well as the capability of producing higher gasoline yield than the reference catalyst. When adopting lanthanum and phosphorus altogether to modify the zeolite Y, a part of La^{3+} can be exchanged with sodium into the super-cage in the zeolite, and the other part can easily react with phosphorus to form superfine complex La–P–O oxides, which obviously precipitate on the exterior surface of zeolite and unavoidably cover part of the surface acid sites.[18] The phosphorus reacted with zeolite Y to form P–OH bonds, and the surface acidity density reduced suitably. Through properly increased acid strength in the pores of zeolite Y, the catalyst effectively controlled the ratio of hydrogen transfer activity and crack ability in the catalytic reaction.

Table 3 – Product distribution of catalyst
Tablica 3 – Distribucija produkata katalize

Product Produkt	Prepared catalyst Sintetizirani katalizator	Reference catalyst Referentni katalizator	Difference Razlika
product yield by mass / % iskorištenje proizvoda po masi / %			
dry gas suhi plin	2.01	2.20	−0.19
LPG	17.43	17.46	−0.03
gasoline benzin	45.77	43.08	+2.69
LCO	17.54	17.85	−0.31
HCO	9.97	11.22	−1.25
coke koks	7.28	8.19	−0.91
conversion konverzija	72.49	70.93	+1.56
light oil lako ulje	63.31	60.93	+2.38
total liquid kapljevine	80.74	78.39	+2.35
product selectivity (yield-to-conversion ratio) selektivnost (omjer iskorištenja i ukupne konverzije)			
dry gas suhi plin	0.028	0.031	−0.003
gasoline benzin	0.63	0.61	+0.02
coke koks	0.10	0.12	−0.02

Table 4 – PONA analyses and gasoline octane number
Tablica 4 – Analiza PONA i oktanski broj benzina

	Prepared catalyst Sintetizirani katalizator	Reference catalyst Referentni katalizator	Difference Razlika
components komponente	volume fraction / % obujamski udjel / %		
normal paraffin normalni parafini	3.7	4.3	−0.6
branched-chain paraffins parafini razgranatog lanca	35.1	32.0	+ 3.1
olefins olefini	16.1	20.7	−4.6
naphthenes nafteni	9.9	10.6	− 0.7
aromatics aromati	35.1	32.4	+ 2.7
octane number oktanski broj			
RON	91.9	91.3	+0.6
MON	81.0	80.5	+0.5

4 Conclusions

A Y zeolite-containing composite with hierarchical porous structure was synthesized by *in situ* technique using polyvinyl alcohol and silica sol material. Due to using polyvinyl alcohol and silica sol, the NaY zeolite had a relative crystallinity of 52.9 % with a high silica/alumina amount ratio of 5.7, and had more micro-, meso- and macro-pores, as well as high specific surface area and pore volume. After modification of lanthanum and phosphorus and steaming stabilization process, a FCC catalyst for maximizing gasoline yield was prepared. Through proper surface acidity and pore modifications, the acidity strength and pore distribution of the catalyst had improved effectively. The catalyst had a wide pore structure and more mesopores distribution. The acidity was more concentrated in the range of intermediate and strong acidity. The evaluated results indicated the catalyst could increase gasoline yield by 2.69 % points and decrease the coke yield by 0.91 % points, while maintaining a high bottom oil cracking ability.

List of abbreviations and symbols
Popis kratica i simbola

BET – Brunauer–Emmett–Teller

BJH – Barrett–Joyner–Halenda

FCC – fluid catalytic cracking
– katalitičko krekiranje u fluidiziranom sloju

HCO – heavy cycle oil
– teško cikličko ulje

LCO – light cycle oil
– lako cikličko ulje

LPG – liquefied petroleum gas
– ukapljeni naftni plin

MON – motor octane number
– motorni oktanski broj

PONA – paraffins, olefins, naphthenes, aromatics
– parafini, olefini, nafteni, aromati

P-RE-USY – ultrastable zeolite Y modified with rare earth elements and phosphorus
– ultrastabilni zeolit Y modificiran elementima rijetkih zemalja i fosforom

RE – rare earth element
– element rijetkih zemalja

RON – research octane number
– istraživački oktanski broj

VGO – vacuum gas oil
– vakuumsko plinsko ulje

VTB – vacuum tower bottom
– ostatak vakuumske destilacije

WHSV – weight hourly space velocity, h^{-1}
– masena prostorna brzina, h^{-1}

Y – zeolite Y
– zeolit Y

w – mass fraction, %
– maseni udjel, %

θ – diffraction angle, °
– difrakcijski kut, °

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SAŽETAK

Povećanje iskorištenja benzina katalizatorom za krekiranje u fluidiziranom sloju (FCC)

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Zeolit Y velikih pora sintetiziran je *in situ* upotrebom polivinilnog alkohola i sola silicijeva dioksida. Za krekiranje u fluidiziranom sloju (FCC) razvijen je katalizator promijenjena sastava.

Zeolit Y u natrijskom obliku imao je relativnu kristalnost 52,9 % i visoki množinski omjer SiO_2/Al_2O_3 5,7. Katalizator s elementima rijetkih zemalja, fosforom i stabiliziran izlaganjem pari, prema analizi FTIR-om, imao je više aktivnih mjesta srednje i jake kiselosti. S takvim preinačavanjem katalizatora više ugljikovodika ulazi u pore i povećava se proizvodnja benzina krekiranjem. Analiza adsorpcijom i desorpcijom dušika pokazuje da katalizator ima više mezo- i makropora što može olakšati prijenos materijala i ubrzati difuziju molekula produkata. Rezultati pokazuju da bi pripravljeni katalizator mogao smanjiti pretjerano krekiranje srednjeg destilata i povećati iskorištenje s obzirom na benzin. Iskorištenje benzina po masi povećalo se za 2,69 %, a iskorištenje koksa i suhog plina smanjilo za 0,91 i 0,19 %. Obujamski udjel olefina u benzinu smanjio se za 4,6 % dok su istraživački oktanski broj (RON) i motorni oktanski broj porasli za 0,6 i 0,5. Dobra selektivnost s obzirom na produkte i visoka iskorištenja benzina u katalizi s pripravljenim katalizatorom očito su povezana sa širokoporoznom strukturom i optimiranom distribucijom kiselosti.

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