Comprehensive Utilization of Filter Residue from the Preparation Process of Zeolite-Based Catalysts

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‖ Abstract
A novel utilization method of filter residue from the preparation process of zeolite-based catalysts was investigated. Y zeolite and a fluid catalytic cracking (FCC) catalyst were synthesized from filter residue. Compared to the Y zeolite synthesized by the conventional method, the Y zeolite synthesized from filter residue exhibited better thermal stability. The catalyst possessed wide-pore distribution. In addition, the pore volume, specific surface area, attrition resistance were superior to those of the reference catalyst. The yields of gasoline and light oil increased by 1.93 and 1.48 %, respectively. At the same time, the coke yield decreased by 0.41 %. The catalyst exhibited better gasoline and coke selectivity. The quality of the cracked gasoline had been improved.

‖ Keywords
Filter residue, catalyst factory, Y zeolite, FCC catalyst, utilization

1. Introduction

The main raw materials for the production of molecular sieve FCC catalyst include water glass, sodium hydroxide, aluminium hydroxide, kaolin, sulphuric acid, hydrochloric acid, liquid ammonia, ammonium chloride, and chlorinated rare earth, alkali, salt, *etc*. The molecular sieve FCC catalyst is prepared through three processes that involve synthesis of molecular sieve, modification of molecular sieve, and preparation of catalyst. During the above processes, a large amount of filter residue is produced. The filter residue is a mixture of amorphous silica–aluminas, zeolites Y and ZSM-5, composed mainly of silica, alumina, Re_2O_3, Na_2O, Fe_2O_3. The moisture content in the filter residue is above 70 %. Compared to kaolin, the filter residue has larger pore volume, specific surface area.

Zeolite-based catalysts manufacturers generate tens of thousands tons of catalyst filter residue each year during production, causing environmental problems that are difficult to handle. Because the filter residue is an appropriate raw material for high-value silicon/aluminium products from either crystal phase composition or main chemical elements, the method for the utilization of the filter residue was developed.[1–4] Application of the filter residue was limited to extracting useful components, such as extracting active SiO_2 and Al_2O_3. The filter residue is predominantly used in the field of construction materials, such as filler for asphalt, and production of bricks and cement. NaX and 4A zeolite were also synthesized using these solid wastes.

The FCC catalyst is probably the most significant, mass-produced modern catalyst.[5–6] The Faujasite type Y zeolite (FAU) has been widely used in the FCC catalyst as the main active component due to its high surface area, large pore size, and proper active sites.[7–10]

The *in situ* synthesised zeolites have an ideal hydrothermal stability. *In situ* synthesizing of zeolite can surmount the difficulty of filtration in industrial production and reduce production costs. Because the filter residue has active useful components, these components can be used for the synthesis of zeolite. We envision synthesizing zeolite Y *via in situ* crystallization technology that enlarges utilization catalogues and quantities of industrial filter residue.

Herein, Y zeolite and an FCC catalyst were synthesized from filter residue. The physicochemical properties of synthesized zeolite and the cracking activity of the catalyst were investigated.

2. Experimental

2.1 Materials

Catalyst filter residue (CFR) was obtained from the Catalyst Factory of Sinopec in China. The filter residue was white gel. Moisture content was 77.8 %. The properties of CFR

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are listed in Table 1 (the composition of compounds and mixtures is expressed in terms of mass fraction).

Table 1 – Properties of catalyst filter residue (CFR)
Tablica 1 – Svojstva filtarskog ostatka katalizatora

Chemical composition Kemijski sastav	$w(SiO_2)/\%$	65.80
	$w(Al_2O_3)/\%$	23.84
	$w(Fe_2O_3)/\%$	0.18
	$w(Na_2O)/\%$	2.15
	$w(Re_2O_3)/\%$	1.44
BET specific surface area/$m^2 g^{-1}$ specifična ploština površine po BET-u/$m^2 g^{-1}$		300
total pore volume/ml g^{-1} specifični obujam pora/ml g^{-1}		0.91
average pore diameter/nm prosječni promjer pora/nm		10.20

Sodium silicate (containing 22.6 % SiO_2, 7.2 % Na_2O) and sodium metaaluminate (containing 20.5 % Na_2O, 3.1 % Al_2O_3) were obtained from *Hunan Juli Catalyst Co., Ltd.*

2.2 Zeolite synthesis

A mixture of sodium silicate, sodium hydroxide, zeolite initiator (the mass ratio of oxides Na_2O, Al_2O_3, SiO_2, H_2O is 16 : 1 : 15 : 320) and filter residue was mixed in sequence. The synthesis of zeolite Y involved an inorganic reaction with a stoichiometry of $Na_2O : SiO_2 : Al_2O_3 : H_2O$ = 2.9 : 1 : 4.8 : 110. The mixture was agitated for 1 h followed by crystallization in a reactor, and heated to 95 °C for 16 – 20 h. The obtained solid product (YZ) was filtered and washed until the pH value of filtered water dropped to 9, and then oven-dried at 120 °C for 4 h. YZ was exchanged one or more times with approximately 10 to 30 % (in mass) of ammonium chloride to replace the sodium, and then exchanged with 3 % (in mass) of lanthanum chloride, followed by calcination at 600 °C with 100 % steam for 2 h to produce modified YZ.

The reference NaY zeolite synthesized by the conventional method involved the following steps: A reaction mixture was prepared by mixing the zeolite initiator with sodium silicate, and adding sodium aluminate solution. The mixture was stirred vigorously, and an aluminium sulphate solution was added to the mixture. The mixture was then heated at a temperature of 95 °C. After 28 hours, the product was filtered and washed. The X-ray diffraction analysis showed an excellent pattern characteristic of zeolite Y.

2.3 Catalyst preparation

Deionized water, kaolin, pseudo-boehmite were mixed with a hydrochloric acid solution and stirred at a rate sufficient to form a uniform slurry. The mixture was stirred for 2 h. Alumina sol and modified YZ were subsequently added, the mixture was stirred at room temperature for 1 – 2 h. The mass ratio of deionized water, kaolin, pseudo-boehmite, alumina sol and modified YZ was 200 : 40 : 13 : 12 : 35. The catalyst (CAT) with a particle size of 0 – 150 μm was produced by spray drying, then calcined at 500 °C for 0.5 h, washed with warm water, and dried.

The reference catalyst (RCAT) was a residue FCC catalyst obtained from a domestic refinery with a specific surface area of 283 $m^2 g^{-1}$ and a specific pore volume of 0.22 $cm^3 g^{-1}$.[11]

2.4 Sample characterization

Relative crystallinity, silica/alumina, crystalline unit cell size and phase of samples were recorded on a *Rigaku Ultimi IV* diffractometer using Cu-Kα radiation (λ = 1.54056 Å) at an operating voltage and current of 40 kV and 30 mA, respectively. The samples were scanned at 0.2 ° min^{-1}. The IR spectra were recorded on an *AVATAR 370 FT-IR* spectrometer in the range from 400 to 2000 cm^{-1}. The acidity properties of samples were determined using pyridine adsorption method. The FTIR spectra exhibited peaks at 1540 and 1450 cm^{-1}, suggesting the existence of Brønsted acid sites, and Lewis acid sites, respectively. The acid strength distributions were quantitatively calculated from the pyridine adsorbed IR spectra at 200 and 400 °C (representing total acid amount and strong acid amount, respectively).[8] The morphology and size of the samples were determined using scanning electron microscopy (SEM) (JEOL JSM-6360) after coating with an Au-evaporated film. The specific surface areas, specific pore volumes, and pore size distributions were measured on an *ASAP 2020* sorptometer using adsorption and desorption isotherm plots at 77 K. A *Malvern Micro-P* particle-size distribution analyser was used for determining the size distribution in the samples. The attrition index of the catalyst was determined using an attrition index analyser by the air injection method.

2.5 Catalytic properties evaluation

The catalytic performance of the catalyst was evaluated on an advanced catalyst evaluation bench unit (ACE, Kayser Corp.). The evaluation conditions were as follows: reactor temperature of 520 °C, catalyst-to-oil mass ratio of 6.0 and WHSV of 19 h^{-1}. Prior to evaluation, the catalyst was steam-deactivated at 800 °C for 17 h with 100 % steam. The feedstock oil consisted of a mixture of 70 % vacuum gas oil (VGO) and 30 % vacuum tower bottom (VTB), and its properties are listed in Table 2.

Table 2 – Properties of feedstock oil
Tablica 2 – Svojstva sirovine za FCC

Variable Veličina	Value Vrijednost
density (20 °C) / g cm⁻³ gustoća (20 °C) / g cm⁻³	0.9222
viscosity (50 °C) / mm² s⁻¹ viskoznost (50 °C) / mm² s⁻¹	79.48
Conradson carbon residue / % Conradsonov koksni ostatak / %	3.8
distillation fraction destilacijska frakcija	temperature / °C temperatura / °C
initial point početna točka	286
10 %	350
30 %	401
50 %	455
70 %	500
90 %	556
end point krajnja točka	>556

Fig. 2 – SEM images of (a) NaY, (b) YZ
Slika 2 – Slike SEM (a) NaY, (b) YZ

Fig. 3 shows the nitrogen adsorption–desorption isotherms and pore-size distribution of YZ. Typical type IV isotherms of YZ confirmed the presence of mesopores and macropores. YZ possessed a wide pore distribution, the distribution was concentrated on approximately 3.0, 4.0, and 5–10 nm, respectively. The total specific surface area of YZ reached a maximum of 640 m² g⁻¹ and its external specific surface area was 76 m² g⁻¹ (Table 3). The fraction of the external surface area in total surface area was 11.9 % (Table 3), which was much larger than that of Y zeolite synthesized by the conventional method, its fraction of external surface area in total surface area was only 4.4 %.

3. Results and discussion

3.1 Structure of zeolite

The chemical composition of CFR was mainly composed of SiO_2 and Al_2O_3, and had higher specific surface area and specific pore volume (Table 1). Using this CFR as raw material, the typical zeolite Y can be obtained. The intensity and shape of the peaks in Fig. 1 indicated that the sample was pure zeolite Y. Under the optimum conditions, the synthesized Y zeolite (YZ) had a relative crystallinity of 71.5 %, and a silica/alumina amount ratio of 5.7, respectively. Fig. 2 displayed that the sizes of the Y zeolite particles were 0.2–0.5 μm, which indicated that smaller crystals had agglomerated with the larger particles.
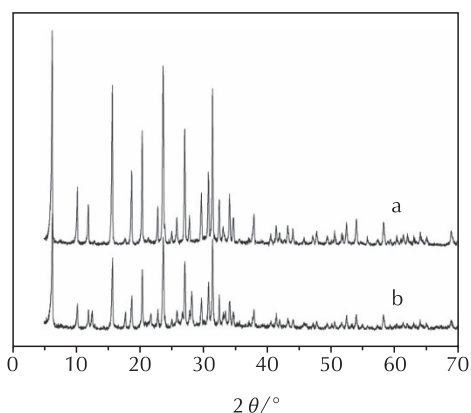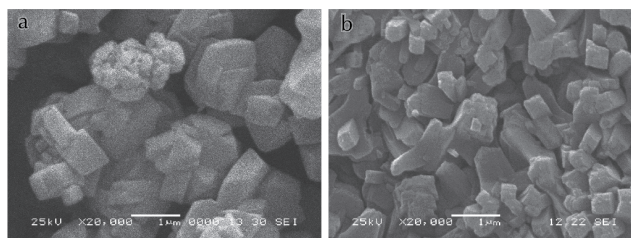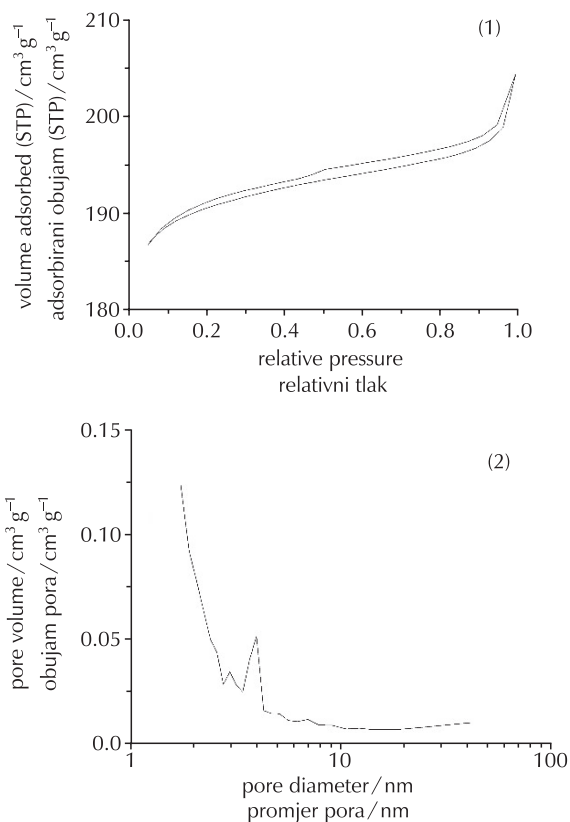

Fig. 1 – XRD patterns of (a) NaY, (b) YZ
Slika 1 – Rendgenski difraktogrami (a) NaY, (b) YZ

Fig. 3 – Nitrogen adsorption-desorption isotherms (1), and pore size distribution (2) of YZ
Slika 3 – Adsorpcijsko-desorpcijske izoterme dušika (1) i distribucija veličina pora (2) zeolita YZ

Table 3 – Properties of YZ and NaY
Tablica 3 – Svojstva zeolita YZ i NaY

Property Svojstvo	YZ	NaY
BET specific surface area/$m^2 g^{-1}$ specifična ploština površine po BET-u/$m^2 g^{-1}$	640	722
BJH specific surface area/$m^2 g^{-1}$ specifična ploština površine po BJH-u/$m^2 g^{-1}$	88	47
micropore specific surface area/$m^2 g^{-1}$ specifična ploština površine mikropora/$m^2 g^{-1}$	564	690
external specific surface area/$m^2 g^{-1}$ vanjska specifična ploština površine/$m^2 g^{-1}$	76	32
total pore volume/$ml g^{-1}$ specifični ukupni obujam pora/$ml g^{-1}$	0.38	0.34
micropore pore volume/$ml g^{-1}$ specifični obujam mikropora/$ml g^{-1}$	0.29	0.31
BJH pore volume/$ml g^{-1}$ obujam BJH pora/$ml g^{-1}$	0.10	0.036
average pore diameter/nm prosječni promjer pora/nm	3.86	2.71
unit cell size/nm veličina jedinične ćelije/nm	2.472	2.468

Fig. 4 shows TG-DTA spectra of YZ. When calcination temperature was up to 920 °C, the skeleton of YZ collapsed, while skeleton collapse occurred at about 850 °C for zeolite synthesized by the common route.[12] YZ exhibited high thermal stability. The main reason is that the YZ zeolite led to wide distribution of particles, which reduced the instability due to low coordination of zeolite synthesized by the common route.[13]
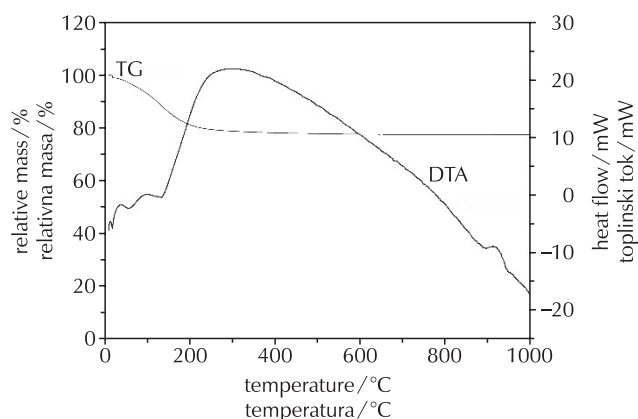

Fig. 4 – TG-DTA spectra of YZ
Slika 4 – TG-DTA-spektar zeolita YZ

3.2 Characterization of catalyst

The properties of the catalyst and the reference catalyst are summarized in Table 4. Comparably, the catalyst has a much larger BET surface area, micropore surface area, total pore volume, and average pore diameter. There were more surface active atoms on the surface compared to the reference catalyst. Therefore, reactant molecules easily get close to the active sites, and thus improve the utilization rate of active sites, leading to higher conversion by the catalysts.

Table 4 – Properties of CAT and RCAT
Tablica 4 – Svojstva katalizatora CAT i RCAT

Property Svojstvo		CAT	RCAT
Chemical composition Kemijski sastav	$w(Na_2O)/\%$	0.15	0.16
	$w(Al_2O_3)/\%$	45.8	50.2
	$w(Fe_2O_3)/\%$	0.26	0.31
	$w(Re_2O_3)/\%$	3.1	3.9
bulk density/$g cm^{-3}$ gustoća/$g cm^{-3}$		0.73	0.75
attrition index/% atricijski indeks/%		1.3	1.5
BET specific surface area/$m^2 g^{-1}$ specifična ploština površine po BET-u/$m^2 g^{-1}$		371	283
BJH specific surface area/$m^2 g^{-1}$ specifična ploština površine po BJH-u/$m^2 g^{-1}$		143	136
micropore specific surface area/$m^2 g^{-1}$ specifična ploština površine mikropora/$m^2 g^{-1}$		274	189
external specific surface area/$m^2 g^{-1}$ vanjska specifična ploština površine/$m^2 g^{-1}$		97	94
total pore volume/$ml g^{-1}$ specifični ukupni obujam pora/$ml g^{-1}$		0.35	0.22
micropore pore volume/$ml g^{-1}$ specifični obujam mikropora/$ml g^{-1}$		0.18	0.10
BJH pore volume/$ml g^{-1}$ obujam pora po BJH-u/$ml g^{-1}$		0.20	0.14
average pore diameter/nm prosječni promjer pora/nm		5.30	4.20
unit cell size/nm veličina jedinične ćelije/nm		2.460	2.456

Fig. 5 shows the nitrogen adsorption–desorption isotherms and pore-size distribution of CAT and RCAT. In Fig. 5 (1),

the isotherms of CAT had a steeper drop and a larger hysteresis loop than RCAT, indicating that larger pores existed in CAT. In Fig. 5 (2), the distribution of CAT appeared from approximately 4.0 to 100 nm, a broad distribution was observed in the pore diameter range > 60 nm, signifying the existence of macropores, and RCAT was observed at approximately 4.0 nm. These results confirmed the hierarchical pore structure of CAT. The catalysts with hierarchical pore structure have the advantages of microporous materials and meso-macroporous materials. Hierarchical materials possessing at least two levels of porosity can reduce diffusion limitations in reactions catalysed by microporous zeolites, which have good selectivity and mass transfer capability.[14]
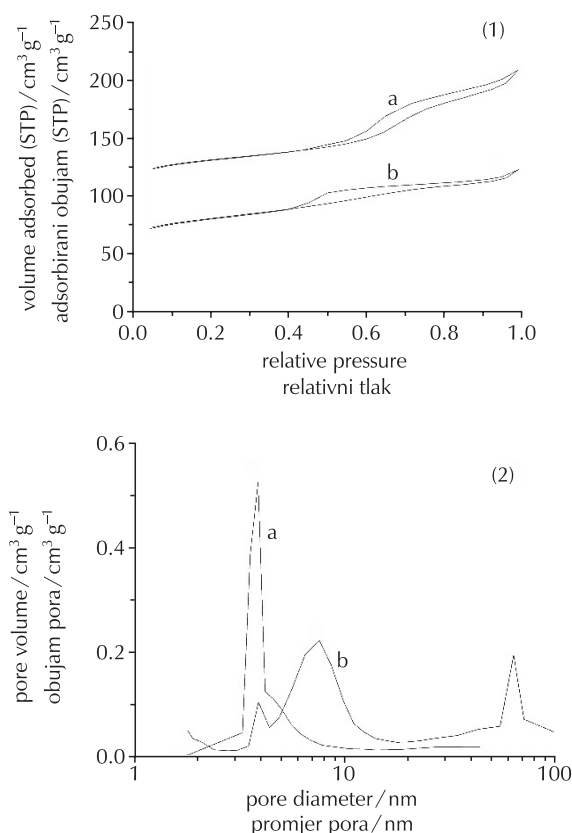

Fig. 5 – Nitrogen adsorption-desorption isotherms (1), and pore size distribution (2) for (a) CAT, and (b) RCAT
Slika 5 – Adsorpcijsko-desorpcijske izoterme dušika (1) i distribucija veličina pora (2) katalizatora (a) CAT i (b) RCAT

The determined acidities of CAT and RCAT are listed in Table 4 and Fig. 6. Comparably, the strong Brønsted acid and the weak Lewis acid amounts of CAT were higher. The more Brønsted acid sites are ascribed to the better crystal structure of Y zeolite synthesized from filter residue. Weak Lewis acid sites and higher Brønsted acid sites are helpful in improving catalytic performance.[15–16]

Table 5 – Acidic properties of CAT and RCAT (relative strength of absorbance bands)
Tablica 5 – Kiselinska svojstva katalizatora CAT i RCAT (relativna jakost apsorpcijskih signala)

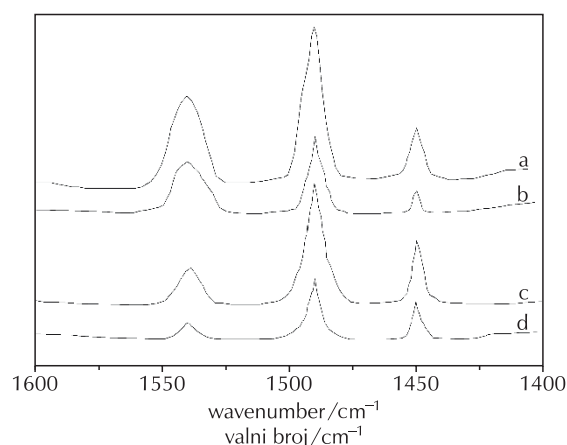| Samples Uzorci	CAT		RCAT	
	Brønsted	Lewis	Brønsted	Lewis
weak acid amount slabo kisela aktivna mjesta	10.4	9.2	7.6	8.5
strong acid amount jako kisela aktivna mjesta	9.3	7.9	4.5	8.2
total acid amount ukupna kisela aktivna mjesta	19.7	17.1	12.1	16.7

Fig. 6 – FTIR spectra of: (a) CAT desorbed at 200 °C, (b) CAT desorbed at 400 °C, (c) RCAT desorbed at 200 °C, and (d) RCAT desorbed at 400 °C
Slika 6 – FTIR-spektri: (a) CAT desorbiran pri 200 °C, (b) CAT desorbiran pri 400 °C, (c) RCAT desorbiran pri 200 °C i (d) RCAT desorbiran pri 400 °C

3.3 Catalytic properties evaluation

The results of catalytic performance for CAT and RCAT are shown in Table 6. It can be seen that compared with the RCAT, the CAT can increase gasoline yield, conversion and liquid yields (LPG + gasoline + LCO) by 1.93, 2.27 and 2.29 %, respectively. At the same time, coke yield is decreased by 0.41 %. Table 7 shows the cracked gasoline compositions over the two catalyst. Comparably, the olefin content of CAT reduced by 2.98 % with the research octane number, and motor octane number of gasoline increased by 0.7 and 0.5. These excellent catalytic performances can be attributed to the novel utilization technology of industrial filter residue.

Table 6 – Product distribution of CAT
Tablica 6 – Proizvodna distribucija CAT-a

Product Proizvod	CAT	RCAT	Difference Razlika
product yield by mass/% iskorištenje proizvoda po masi/%			
dry gas suhi plin	2.17	2.23	−0.06
LPG	15.43	14.62	+0.81
C$_{5+}$ gasoline benzin C$_{5+}$	47.71	45.78	+1.93
LCO	18.80	19.25	−0.45
heavy oil teško ulje	8.95	10.77	−1.82
coke koks	6.94	7.35	−0.41
conversion konverzija	72.25	69.98	+2.27
light oil lako ulje	66.51	65.03	+1.48
total liquid ukupno kapljevine	81.94	79.65	+2.29
product selectivity (yield-to-conversion ratio) selektivnost (omjer iskorištenja i ukupne konverzije)			
dry gas suhi plin	0.030	0.032	−0.002
gasoline benzin	0.66	0.65	+0.01
coke koks	0.096	0.10	−0.004

Table 7 – PONA analyses and gasoline octane number
Tablica 7 – Analiza PONA i oktanski broj benzina

Product Proizvod	CAT	RCAT	Difference Razlika
components komponente	volume fraction/% obujamski udjel/%		
normal paraffins normalni parafini	3.87	3.95	−0.08
branched-chain paraffins parafini razgranatog lanca	24.91	23.24	+1.67
olefins olefini	19.75	22.73	−2.98
naphthenes nafteni	9.42	9.56	−0.14
aromatics aromati	42.05	40.52	+1.53
	octane number oktanski broj		
research octane number istraživački oktanski broj	91.8	91.1	+0.7
motor octane number motorni oktanski broj	80.9	80.4	+0.5

Conclusion

A new utilization technology of the filter residue has been successfully developed. Zeolite Y was synthesized from filter residue and the characterization results showed that Y zeolite had a relative crystallinity higher than 70 % with a silica/alumina amount ratio of 5.7, as well as better hydrothermal stability. The as-made FCC catalyst had moderate acidity, and an advantageous pore system comprising micropores, mesopores, and macropores, and then showed excellent catalytic performances on activity, bottoms cracking ability, and good yield of valuable products in heavy crude oil processing. In comparison to the reference catalyst, the yields of the gasoline and light oil increased by 1.93 and 1.48 %, respectively, and the olefin content in the cracked gasoline reduced by 2.98 %, while the research octane number increased by 0.7.

List of abbreviations and symbols
Popis kratica i simbola

BET   – Brunauer–Emmett–Teller
BJH   – Barrett-Joyner-Halenda
CAT   – catalyst
       – katalizator
CFR   – catalyst filter residue
       – filtarski ostatak katalizatora
DTA   – differential thermal analysis
       – diferencijalna termička analiza
FAU   – Faujasite
       – Faujasit
FCC   – fluid catalytic cracking
       – katalitičko krekiranje u fluidiziranom sloju
LCO   – light cycle oil
       – lako cikličko ulje
LPG   – liquefied petroleum gas
       – ukapljeni naftni plin
PONA   – paraffins, olefins, naphthenes, aromatics
       – parafini, olefini, nafteni, aromati
RCAT   – reference catalyst
       – referentni katalizator
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}
YZ   – synthesized Y zeolite
       – sintetizirani zeolit Y
TG   – thermogravimetry
       – termogravimetrija

w – mass fraction, %
– maseni udjel, %
θ – diffraction angle, °
– difrakcijski kut, °

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SAŽETAK

Potpuno iskorištavanje taloga preostalog nakon priprave zeolitskog katalizatora

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Proučena je upotreba taloga preostalog nakon priprave katalizatora baziranog na zeolitima. Od taloga su pripravljeni zeolit Y i katalizator za katalitičko krekiranje u fluidiziranom sloju (FCC).

Ovako sintetiziran zeolit Y termički je stabilniji u odnosu na zeolit Y sintetiziran konvencionalnom metodom. Katalizator ima široku distribuciju veličine pora. U odnosu na referentni katalizator pokazuje veći obujam pora i specifičnu površinu te bolju otpornost na atriciju. Iskorištenje benzina i lakog ulja povećalo se za 1,93 i 1,48 %, a koksa smanjilo za 0,41 %. Katalizator je pokazao bolju selektivnost s obzirom na benzin i koks, a poboljšala se kvaliteta krekiranog benzina.

Ključne riječi
Filtarski talog, tvornica katalizatora, zeolit Y, katalizator FCC, iskoristivost

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