Perspective on FCC catalyst in China

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Abstract This paper provides an overview of the enormous challenge in processing heavier fluid catalytic cracking (FCC) feedstock and producing higher qualified liquid fuels. Besides optimizing the operation conditions of the FCC unit, it is crucial to design new catalysts especially for heavier and inferior feedstock. In this paper, a new concept, stepwise structure of catalyst, was postulated and a potential new catalyst based on stepwise structure design was prepared.

Keywords Fluid catalytic cracking · Heavy oil ·
Catalyst · Zeolite

Introduction

Fluid catalytic cracking today is one of the most important technologies for upgrading of heavy hydrocarbon fractions to light liquid fuels in most modern petroleum refineries. It is reported that the installed worldwide FCC capacity is over 2 million tons per day as of 2012, which represents 16.49 % of worldwide crude unit capacity [1, 2]. As an important secondary processing means, the typical feedstock for FCC process are gas oils derived from atmospheric and vacuum fractionators. However, because crude oil supply all over the world is becoming heavier and

bastardized in quality and the demand for middle distillate is larger, the FCC process is now in rapid development.

The lighter crude oil supply is less than the demand from 2003, requiring new and enhanced production of extra heavy oils and eventually tar sands [3]. Nowadays, global residue processing is up to 0.87 billion tons every year, 23.3 % of which is processed by FCC. In China, FCC capacity was over 170 million tons in 2010 and more than 60 % of heavy oil was converted by the FCC process, providing about 75 % gasoline pool, 35 % diesel and 40 % propylene. Besides that, environmental legislation concerning aromatics and sulfur and nitrogen contents in transportation fuels are becoming more and more strict. The situation is even more severe in China due to the increasing dependence on imported crude oil and on the FCC process which produces gasoline with high olefin and high sulfur content. Therefore, it is crucial to change and adjust the FCC process conditions for improving the quality of transportation fuels and protecting the environment.
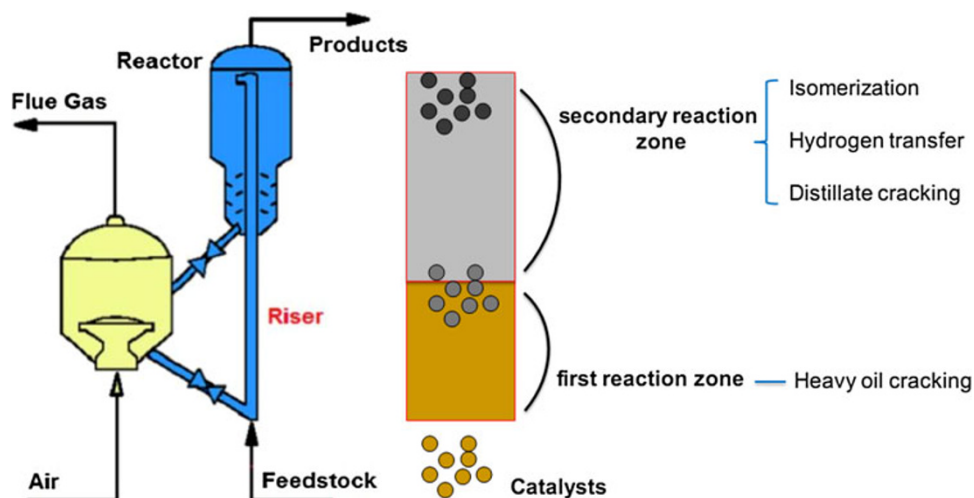
FCC reactions

FCC unit

A typical FCC unit is composed of a reactor and regenerator, distillation column and absorption stabilization system. The reactor and regenerator are considered to be the heart of the fluid catalytic cracking unit. The simplified schematic flow diagram of a typical modern FCC unit in Fig. 1 is based on the "high–low paratactic" configuration. The preheated high-boiling-point petroleum feedstock (at about 315–430 °C), consisting of long-chain hydrocarbon molecules and recycled slurry oil from the bottom of the

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distillation column, is injected into the catalyst riser, where it is vaporized and cracked into smaller molecules of vapor by contact and mixing with the very hot powered catalysts from the regenerator. All of the cracking reactions take place in the riser within a period of 2–4 s or less at a temperature of about 500 °C or higher. The cracked product vapors and spent catalyst then flow upward through a set of cyclones within the reactor and they are further separated through a steam stripping section to remove any hydrocarbon vapors before spent catalysts return to the regenerator. The cracked product vapors flow from the top of the reactor to the bottom section of the distillation column, i.e., fractionators, where they are distilled into the FCC end products of cracked naphtha, fuel oil and off gas. Since the cracking reactions produce some carbonaceous material that is deposited on the catalyst and very quickly reduces the catalyst reactivity, the spent catalyst must be regenerated by burning off the deposited coke with air blown into the regenerator. The combustion of coke is exothermic and very hot (at about 700 °C). It produces a large amount of heat required for the vaporization and endothermic cracking reactions of petroleum feedstock when catalysts are recycled into the catalyst riser. The FCC unit is operated in the steady state. The operation shows no overall change with time. Therefore, the unit must be in energy balance, i.e., the energy released by coke burning in the regenerator must satisfy the heat needed for cracking reactions as well as other energy that exited.
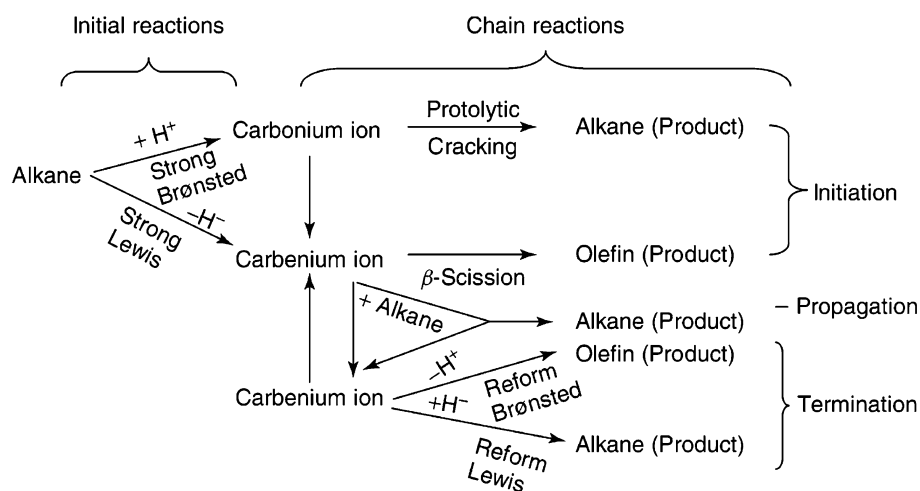
Reaction sections

In commercial FCC operation, the main reactions of preheated feedstock in the catalyst riser are thermal cracking reactions on the surface of regenerated catalysts and catalytic cracking reactions. In these reactions, thermal cracking reactions take place in the bottom section of the riser where the temperature exceeds 600 °C via radical reaction mechanisms. Thermal cracking proceeds by the direct breaking of carbon–carbon bonds, resulting in excessive light gas yield and lower overall product values [4, 5]. Ethylene is the major product and is the chain-terminating product from the thermal cracking of paraffins. Propylene and higher olefins are also produced in decreasing yields with no branching. On the other hand, acid-catalyzed cracking reactions play a main role in the upper section of the riser at lower temperature via carbenium ion mechanism, leading to both olefins and paraffins with branching. Propylene is the chain-terminating product and a high yield of propylene over ethylene is a fingerprint for catalytic cracking compared with thermal cracking [6].

Reaction mechanisms

Catalytic cracking is catalyzed by Brønsted and Lewis acid sites of an acid solid such as SiO_2–Al_2O_3 or zeolites and involve C–C bond breakup via formation of carbocations (carbenium and carbonium ions) transition states. A study of the chemistry of catalytic cracking relies on the rich chemistry of carbocations. The basic reaction chemistries involved in catalytic cracking are discussed in very rich literature [7, 8]. A typical mechanistic scheme for catalytic cracking via carbenium ion intermediates is shown in Fig. 2 [9]; it involves initiation on either a Brønsted or Lewis acid site by protonation or H-abstraction, respectively. Propagation then occurs by hydride transfer and cracking by β-scission. Termination reactions involve reaction of a carbenium ion with either a Brønsted base to form an alkene and a Brønsted acid site, or with a Lewis base to from an alkane and reform a Lewis acid, or with a hydride ion from coke to form an alkane. Initiation and cracking can also occur by reaction of an alkane with a Brønsted acid site to form a carbonium ion, which in turn

Fig. 2 Reaction pathway network for alkane cracking

can react via β-scission to a smaller alkane and a carbenium ion or undergo H₂ elimination to a large carbenium ion.

In addition to the initial cracking reactions to form alkane and olefin, there are a number of secondary acid-catalyzed reactions that occur during hydrocarbon cracking, such as skeletal and/or double-bond position isomerizations, cyclization reactions, aromatization, alkylation, dealkylation, branching and polymerization. The secondary reactions produce a large amount of low carbon molecules such as aromatics and alkenes which are in favor of improving octane number of gasoline and propylene and butane yields. However, overcracking reactions of distillates also decrease light distillate yields and increase dry gas and coke formation, which are usually unexpected. Taking the thermal cracking into consideration, the FCC operation proceeds via parallel series reactions in the catalyst riser, converting high molecule hydrocarbons into light distillates, gases and coke.

Therefore, it is crucial to coordinate thermal cracking and secondary reactions occurring in the FCC process. One industrial successful example is the two-stage riser FCC technology (TSRFCC) proposed by the University of Petroleum (East China). The cracking reactions are reinforced by renewing the catalyst in the second stage of catalyst riser. In a similar conversion level, the TSRFCC has increased gasoline and light oil yields and lower gas yield compared with the one-stage riser (OSR).

FCC operating variables and FCC feedstock

The performance of an FCC unit is dependent on a large number of parameters. The feed composition, residence time, temperature, catalyst-to-oil ratio (CTO), hydrocarbon partial pressure, catalyst properties and riser hydrodynamics influence the conversion process in their own way.

The effects of operating parameters (reaction temperature, CTO, contact time) on thermal cracking and secondary cracking are obvious [10]. Endothermic reactions dominate the whole reactions of the FCC cracking process. Specifically, cracking and dealkylation with C–C bond breakup are endothermic and favored at high temperature, leading to high conversion of heavy hydrocarbons. In contrast, hydrogen transfer, isomerization reactions and polymerization reactions are exothermic reactions and favored at lower temperature. Generally, low reaction temperature, high CTO and short contact time of oil and catalyst may suppress the detrimental thermal cracking dramatically and benefit hydrogen transfer and isomerization reactions. In the overcracking operation caused by high temperature, thermal cracking may be enhanced, and hydrogen transfer and isomerization reactions may be decreased remarkably, while condensation reaction between olefin and aromatic hydrocarbons can be enhanced. In the overcracking operation caused by high catalyst-to-oil ratio, thermal cracking may be restrained, while hydrogen transfer and isomerization reactions in the secondary reactions can be promoted. In the overcracking
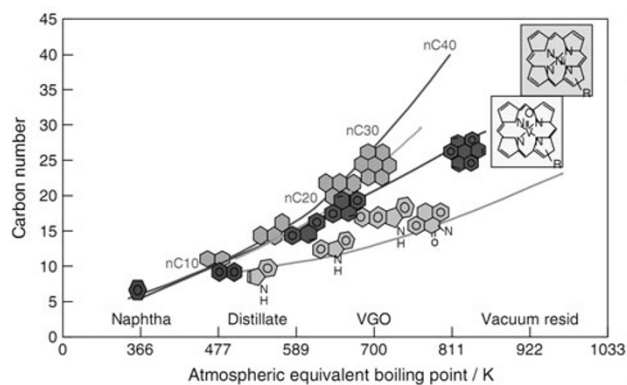
Fig. 3 Molecular distributions of petroleum

operation caused by long contact time, thermal cracking reaction increases and hydrogen transfer and isomerization reactions appear to have the same tendency as in thermal cracking reaction [6].

Generally, FCC feedstocks include paraffins, naphthenes, aromatics and polar compounds, as shown in Fig. 3 [11]. Typical feedstocks contain 50–66 % paraffins, 15–25 % naphthenes, 15–25 % aromatics and <5 % olefins. Paraffins are straight- or branched-chain hydrocarbons which are made up molecules with about 20–40 carbon atoms in the vacuum gas oil (VGO) range. Aromatics are compounds that contain at least one benzene ring, while naphthenes are saturated ring compounds. In general, FCC feedstocks contain some levels of common heteroatoms such as sulfur, nitrogen, nickel and vanadium. The composition of the feedstock influences the quantity and quality of the final products. For example, paraffinic feeds are easy to crack and produce low coke and high gasoline. However, aromatic compounds are more refractory to crack than paraffinic feedstock because they are: (i) lower in H content and (ii) associated with low diffusion coefficients. So these reactions result in lower conversion, lower gasoline and higher coke.

During cracking, the metal compounds from feedstock deposit quantitatively on the catalyst. Nickel and vanadium are particularly noticed because they are present in high concentrations and have the most detrimental effects on the cracking performance of the FCC catalyst. Nickel deposits on the catalyst surface in two primary structures of dispersed nickel oxide and nickel aluminate. The former promotes dehydrogenation reactions, consequently increasing yields of coke and hydrogen while decreasing gasoline production. Vanadium also deposits on the catalyst surface, but unlike nickel migrates through the catalyst and reacts with zeolites and rare-earth metals forming vanadate complexes, which easily destroys the zeolites. Vanadium contamination reduces conversions 3–4 times, reduces gasoline yield about 1.2 times and increases hydrogen and coke yields 0.3–0.4 times as much as nickel [12]. Also, sulfur and nitrogen in the FCC feedstock lead to SO_x and NO_x emissions in the regenerator and into distillates and must be controlled.

The traditional use of FCC converting heavy VGO, i.e., the overhead product from vacuum distillation of crude unit bottoms remains the primary duty. The conventional FCC catalysts are all effective in reducing slurry to some extent. The focus of FCC catalyst research and development is more on matrix to reduce coke and gas formation and/or how to make the active component (zeolite Y) more effective.

However, a growing number of refineries are feeding heavier and bastardized oils (residual oil or resids) along with VGO. In China, the vacuum resids added into FCC feedstock are up to 30 %; this ratio in some refineries is up to 60–70 %. It becomes more difficult to process more resids for producing gasoline and light fuel oil. The properties of resids account for the difficulties, and the reasons are as follows:

(a) Resids have increased molecular weight (MW) and higher boiling point fractions and need more energy to vaporize them to enter the catalyst riser.

(b) Resids with lower hydrogen content and increased asphalthenes, resins, polar compounds and Conradson carbon residue (CCR) are prone to form dry gas and coke.

(c) The increased sulfur and nitrogen contents in resids make it harder to meet environmental legislations.

(d) The increased contaminant metals (Ni, V, Na, Ca, Fe) in resids lead to more dry gas and coke formation, catalyst deactivation and destruction.

Significant variations in the qualities of products occur when resids are proportionally incorporated into the conventional VGO feedstock. The FCC catalysts are easier to deactivate along with higher delta coke formation and metal deposition, leading to reduced conversion of feedstock. Generally, four types of coke form in the FCC process: catalytic coke formed during the cracking reactions; contaminant coke from the activity of contaminant metals such as Ni and V; feed coke from the con-carbon in the feed; cat-to-oil coke from "unstripped" hydrocarbons that carry over into the regenerator. The severe deactivation from coke formation and metal contamination after resid incorporation is unavoidable, as a result dry gas yields including hydrogen and slurry oil yield increase while gasoline selectivity decreases.

In these cases, the spent catalysts cannot be recovered completely in the regenerator. The endothermic capacity needed for cracking reactions of FCC feedstock must be balanced with exothermic capacity when burning coke and energy lost in the flue gas and unit emission. Limited to regenerator temperature and air blasting capacity for coke burning as well as the heat removal from main column, the spent catalysts cannot be recovered to some extent. Besides optimizing FCC operating conditions, another alternative is to design and develop new FCC catalysts for FCC effective conversion.

FCC catalyst design

Composition of FCC catalysts

Modern FCC catalysts provide acid-catalyzed sites for hydrocarbon reactions. They are powered solid acids principally comprising zeolites, matrix, binder and sometimes additives.

Zeolites employed in manufacture of the FCC catalysts are synthetic versions of naturally occurring zeolites called faujasites. Virtually, all of today's catalysts contain Y zeolite or variation thereof. Zeolites possess particular characteristics of crystalline structure and acidity which are responsible for their capability to catalyze chemical reactions. The pore system of Y is characterized by a set of four tetrahedrally oriented 12-membered ring opening leading to a larger central cavity, i.e., supercage. The pore opening is 0.74 nm in diameter, while the supercage is 1.2 nm in diameter. Due to the large number of cationic sites in high surface area zeolite resulting from Si and Al atoms and possibly also due to electric field gradients set up by the cationic sites in the nanopores and cages of zeolites, a large number of acid sites with special concentration and strength are formed. Nevertheless, zeolite acid concentration and strength vary over a wide range, mainly as a function of Si/Al ratio. Generally, the acid site concentration is simply proportional to the inverse of the Si/Al ratio; in contrast, acid site strength varies inversely with Si/Al ratio. However, to modify the stability and acidity and satisfy the requirements of FCC conditions, zeolite Y is typically modified by ion exchange generating acidity, or hydrothermal treatment producing the so-called ultrastable Y (USY), or exchanging with multivalent cations such as rare-earth ions (La^{3+} and Ce^{3+}) for improving acid strength.

Generally, the matrix of the FCC catalysts is everything except active zeolites, including the binder, the clay and functional components. However, during the hydrothermal conditions of commercial FCC unit, about half of the starting zeolite is destroyed and becomes part of the matrix [13]. The overall matrix composition during use is formed by the solid-state reactions of all the non-zeolitic components. Besides the mechanical properties in fluidized state, the matrix added must to a certain extent improve bottom oil cracking, impart metals resistance or provide specialized functions such as lowering sulfur content in gasoline. Commercially, active components based on specialty aluminas are incorporated into most fluid cracking catalysts for increasing the matrix surface area and matrix acidity necessary to precrack the larger molecules into smaller ones that can then enter the pores of the zeolite. The active components also increase the pore volume of the catalyst and allow faster diffusion of resid molecules. The surface area, pore volume and thermal stability of the alumina are important for coke selectivity, bottoms cracking and metal tolerance, as well as to passivate the nickel and vanadium [14, 15]. In addition, clay may be incorporated as matrix or balance of the formulated catalysts and has certain similar functions such as precracking and mechanical strength.

New FCC catalyst design

According to thermal cracking and secondary reactions of heavy hydrocarbons occurring in the FCC process, it is believed that radical reactions for hydrocarbon precracking happen mainly on catalyst matrix at high temperature in the first reaction zone of the catalyst riser. Because chain paraffin reacts to mainly form ethylene, methane and α-olefins by radical reactions, heavy oil cracking on matrix easily forms dry gas and coke in this section. Dominant reactions in catalytic cracking take place in the secondary reaction zone of the catalyst riser. The cracking reactions in this section are based on the carbenium reaction mechanism and dependent on catalyst zeolites. The supercage of zeolite Y is critical to heavy oil cracking for offering acid sites and promoting carbenium ion formation. The unconverted slurry yield decreases remarkably with increasing zeolite Y content of the FCC catalysts.

Fig. 4 Idea on new catalyst design for effective heavy oil cracking
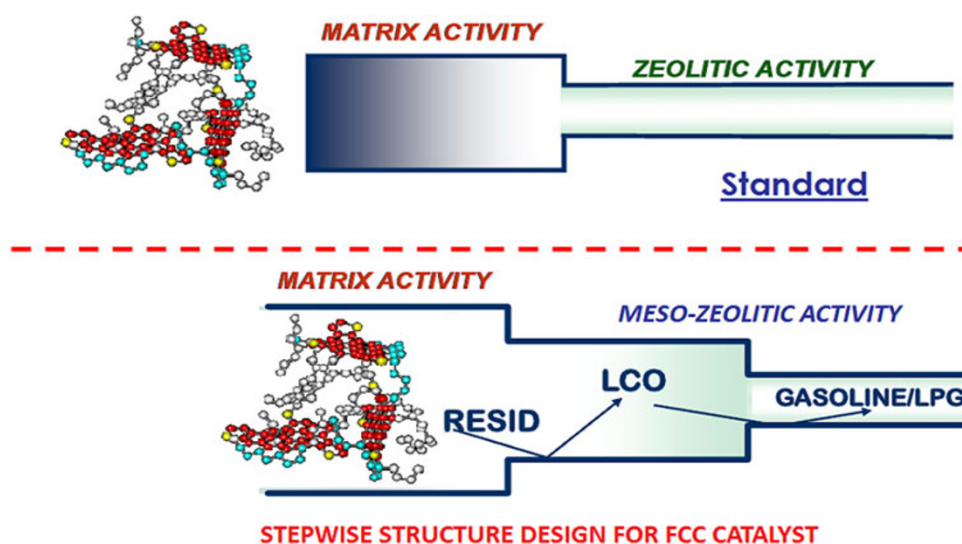
The reaction mechanism discussed above and the philosophy of common catalyst processing FCC feedstock are generally accepted and displayed in Fig. 4. Heavy hydrocarbons first go through precracking reactions mainly on the matrix because of their accessible pores and external/internal surface active sites. These reactions are important for the conversion of FCC feedstock to heavy light oil (HCO), light cycle oil (LCO) as well as dry gases. Simultaneously, small-molecule radical ions or a small number of carbenium ions via radical ion mechanism and carbenium ion mechanism are formed which play a key role in secondary reactions. Thermal cracking is beneficial to some degree for further conversion of feedstock. Secondly, secondary cracking reactions mainly take place in the micropores of zeolites when smaller hydrocarbon is accessible to strong acid sites in the cages of zeolites. In this process, light liquid fuels with boiling point in the gasoline range and small molecule alkenes such as propylene are formed. The reactions of conventional FCC VGO feedstock are well explained by the standard scheme in Fig. 4.

However, cases are different when heavier resids are involved in the FCC feedstock. For VGO feedstock, carbon numbers are 15–30 and the dynamic diameter is smaller than 2.5 nm. The large molecule intermediates derived from precracking reactions of VGO may be accessible to active sites in zeolites (zeolite Y with 0.74 nm). However, vacuum resids have bigger carbon numbers of more than 40 and dynamic diameters of 2.5–15 nm. Most of them are too large to fit into the zeolite pores even through inadequate precracking.

Successful resid fluid cracking catalysts have become a challenge; the new catalysts require enhanced properties as follows:

(a) enhanced bottoms cracking for slurry conversion and low "feed coke";
(b) high tolerance to contaminants and low "metal coke";
(c) optimized hydrogen transfer to limit "catalytic coke" and desired balance between LPG and gasoline;

(d) open pore structure for low "stripping coke", enhanced cracking of large feed molecules and reduced overcracking of desired products.

Therefore, stepwise structure design for the FCC catalyst is introduced for heavier feedstock conversion as shown in Fig. 4. In this stage, meso-zeolites with both micro- and meso-pores are introduced into the FCC catalysts. Mesopores of zeolites are favorable for further secondary cracking reactions of large molecule intermediates. This design is beneficial for the conversion of FCC feedstock when resids are added. Simultaneously, liquid yields of gasoline and diesel increase due to limited secondary overcracking of gasoline to liquefied petroleum gas (LPG). Zeolites Y with both micropores and mesopores can be prepared in many ways, such as template method, acid or alkali post-treatment, hydrothermal treatment and so on. Modification of matrix is also used to lower matrix activity and widen pores.

The design philosophy of new catalysts is based on the idea of off-site cracking for heavy oil. A little big feed molecules precrack on matrix to produce small fraction hydrocarbon, which is feasible to enter into the supercage of zeolite Y and obtain H^+ released by Brønsted acid site to form carbeniums. Some carbeniums can go through β-scission to become smaller; however, the others diffuse out of the supercage. The carbeniums that diffuse outside encounter heavy molecules to form huge carbeniums, which leads to crack through β-scission and it is important for heavy oil cracking. Such a cracking is remarkably different form adsorption–desorption reaction that takes place on the active sites of catalysts, not resulting in coke formation, but realizing effective heavy oil cracking.

Therefore, continuous intracrystalline production of carbeniums and delivery of carbenium outside are crucial for effective cracking of heavy oil. Specifically, catalysts are designed in three ways: to advance matrix, to improve accessibility with high active center accessibility and to prepare stable zeolites. The new matrix should have lower

Table 1 Some industrial FCC catalysts for heavy oil in China

Model	R&D comp.	Active components	Applications
DVR-1 DVR-2	RIPP and catalyst complex of Qilu Petrochemical Corporation	Modified USY and REHY	Used for full VR in Daqing Petrochemical Corporation
LVR-60	RIPP	Modified USY and REHY	Used for high resids addition ratio feedstock
OREBIT-3600	RIPP and catalyst complex of Qilu Petrochemical Corporation	USY, REHY, modified Al_2O_3	Tolerance to metal performance (Ni, V)
CHV-1	RIPP and Luoyan Petrochemical Complex and Changling oil refining chemical factory	SRY (USY), REUSY	Tolerance to metal performance (Ni, V)
RAG-7	RIPP	ZSM-5	High LPG yield and high-ON gasoline

activity, reducing coke formation as much as possible. In addition, it should have highly porous structure, enhancing the thermal and hydrothermal stability and increasing the chance of contact of heavy oil molecule and carbeniums. Zeolite Y should be with high stability, high molecule accessibility (i.e., Albemarle Accessibility Index, AAI), activity maintenance and cracking selectivity.

In China, research and applications of FCC catalysts for heavy oil are popular. Table 1 gives some industrial FCC catalysts specially for processing feedstock with resid addition. All these catalysts are designed for some special requirements like increasing the conversion of heavy oil and light oil yields, decreasing coke yield, enhancing resistance to metal pollution performance and so on. However, companies differ on the development of catalysts. For instance, DVR catalysts can especially increase the cracking of Daqing full vacuum resid as well as decrease olefin content in FCC gasoline. CHV catalysts consist of zeolite with high SiO_2/Al_2O_3 ratio and have good performance in coke selectivity and isomerization reactions; at the same time these are tolerant to vanadium pollution. In addition, new FCC catalysts especially for processing of heavier FCC feedstock have been developed in China. Based on the design philosophy of the aforementioned new catalysts, the newly designed active component (i.e., zeolite) has higher thermal and hydrothermal stabilities compared with commercial zeolite. Additionally, new matrix shows weaker acidity and more open pore structures which is in favor of the diffusion of carbonium ions. The performances of this catalyst in a laboratory riser and in a pilot riser are displayed in Tables 2 and 3. Both show that new catalysts have better performance in improving conversion of feedstock and total yields, while reducing slurry and coke yields than industrial catalysts. Naphtha has high octane number but decreased olefins, beneficial for improving the quality of FCC gasoline.

Table 2 Performance of new catalyst in laboratory riser

Items	Domestic comp.	New catalyst	Foreign comp.
Dry gas, wt%	1.30	1.46	1.21
LPG, wt%	18.76	19.45	15.39
Naphtha, wt%	48.47	48.73	51.52
LCO, wt%	15.93	17.14	16.47
Slurry, wt%	7.63	4.77	6.09
Coke, wt%	7.56	8.31	8.81
Total yields, wt%	83.16	85.32	83.58
Olefins in naphtha, %	46.39	37.84	44.95
GC-RON	94.2	94.2	92.2

Table 3 Performance of new catalyst in pilot riser

Items	Blank	Final trial	Δ
Dry gas, wt%	3.64	3.14	−0.50
LPG, wt%	12.83	13.07	0.24
Naphtha, wt%	45.15	47.43	2.28
LCO, wt%	26.20	25.37	−0.83
Slurry, wt%	4.61	4.26	−0.35
Coke, wt%	7.57	6.91	−0.66
Total yields, wt%	84.18	85.69	1.51
GC-RON	88.6	90.3	1.7

Conclusions

FCC plays a critical role in China's commercial supplies of vehicular fuel and chemicals. To effectively process heavier FCC feedstock with conventional VGO and more inferior resids, a new catalyst with stepwise structure was postulated and prepared. This new catalyst is composed of matrix and zeolites with stepwise porosity and acidity that allow stepwise diffusion and cracking of heavy molecules while minimizing coke formation.

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