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Heavy naphtha upgrading by catalytic reforming over novel bi-functional zeolite catalyst

Nada S. Ahmedzeki[1] · Ban A. Al-Tabbakh[2] · Maher B. Antwan[2] · Selahattin Yilmaz[3]

Abstract

Naphtha catalytic reforming process using bimetallic platinum and titanium loaded on nano synthesized HY zeolite was investigated for the product octane number enhancement. The activity of five samples of the prepared catalyst with different bimetallic loading of 0.13, 0.25 wt% Pt and 0.75, 1, 2.2 wt% Ti were investigated for Iraqi heavy naphtha catalytic reforming at the temperature range of 490, 500 and 510 °C and pressure of 10 bar in a packed bed pilot plant reactor. The best result of the research octane number was 84 for reformate product using the sample of 0.13% Pt–0.75% Ti%/HY zeolite at temperature 510 °C and 10 bar achieving 47% increase. All samples of the prepared catalyst showed a good stability during the operation at severe conditions and the maximum carbon content was about 9% due to the deposition of coke precursor on the catalyst surface. The reformate yield was investigated for all catalyst samples under 10 and 20 bars and the same range of temperature and ranged from 89 to 94%. These results are encouraging for future possible replacement of the conventional catalyst by the local synthetic zeolite. Octane number was correlated on the basis of constituent's boiling points. The maximum absolute error between the experimental and predicted octane number was about 3 and 5% using 10 and 20 bar respectively.

Keywords Catalytic reforming · HY catalyst · Bi-functional catalyst · Octane number enhancement

✉ Nada S. Ahmedzeki
dr_ahmedzeki@yahoo.com

Extended author information available on the last page of the article

Introduction

The catalyst is the heart of the reforming process and most of the major improvement in the process was due to the development of improved catalyst. Since its beginning in 1940 molybdenum—oxide was supported on alumina catalyst in fixed bed reactors with very short time between regenerations to burn of deposited carbon [1]. Later in 1950 this catalyst was used in a fluid bed process with continuous regeneration to decrease the cycle times and increase the catalyst activity. UOP (Universal Oil Products) was first to introduce platinum—alumina catalyst (platforming process) in 1948. Platinum is more stable than the one previously used and this catalyst provided the higher reformer severities needed to meet the increasing motor gasoline octane requirements. Chevron research announced in 1969 its rhenium promoted Pt/Al_2O_3 reforming catalyst and the effect of rhenium is to lower the rate at which carbon deposited on the catalyst surface [2]. So catalytic reforming is the fastest growing of all refining processes and many investigations are carried out for different aspect of catalytic reforming process and focused on three important issues; studying and creating new catalysts with better activity, selectivity as well as deactivation, investigating the reaction of reforming and disclosing suitable kinetics and deactivation models and, presenting or offering reactor configuration and mode of operation with higher performance and yield.

The catalytic reforming process encompasses actually a complex network of reactions and the four major reactions are: (i) Dehydrogenation of Naphthenes (ii) Isomerization of Paraffins and Naphthenes (iii) Dehydrocyclization of Paraffins (iv) Hydro Cracking and Dealkylation [3]. The overall net production of hydrogen in the catalytic reforming of petroleum naphtha range from about 50–200 cubic meter of hydrogen at (0 °C, 1 atm.) per cubic meter of liquid naphtha feed stock. This hydrogen may be used elsewhere in the refinery plant, for example, in the hydrodesulfurization process [4, 5]. Naphtha reforming units are usually classified according to the catalyst regeneration procedure: (i) Semi-regenerative catalyst reformer (SRR) (ii) cyclic catalytic reformer (iii) continuous catalyst regeneration Reformer (CCR) [4].

The most commonly used type of the reforming process is the semi—regenerative catalytic reformer where about 60% worldwide is using this process. This process is identified by long period of continuous operation with decreasing catalyst due to coke. It is generally built with a series of catalyst bed reactors operated at a temperature range from 495 to 520 °C and pressure of about 5–45 atm with research octane number of 85–100. Several points are watched due to regeneration such as reactor metallurgy temperature, specific amount of $C5^+$ yield decline and specific amount of hydrogen decline. Shut down of the units occurs once each 6–24 months [6]. In the cyclic reformer, each reactor may be undergoing a wide boiling range feed, low operational pressure, low hydrogen to feed ratio, and catalysts are exhausted within 1 week to month. The octane number is about 100–104. All reactors are operated between reducing atmosphere during normal operation and oxidizing atmosphere during regeneration. This switching policy

needs a complex process layout with high safely precautions. This process is not very common and is rarely used [4].

Continuous catalyst regeneration reformer is the most modern type of catalytic reformer. The catalyst is regenerated continuously in special regenerator and this process has many advantages towards traditional ones because of the following reasons: Higher octane reformate is obtained even working with low feed quality, catalyst with less stability but higher yield and selectivity are achieved and, low recycle ratio is required with higher hydrogen yield. Reactors are placed separately or stacked on each other and the catalyst moves from the bottom to regenerator then moves to the top of first reactor. In these reactors sophisticated reactions take place; converting Naphthenes to aromatics takes place in the first reactor and the outlet from first reactor is reheated and fed to the second reactor where the major reaction is isomerization. After the preheating of the outlet of second reactor it will be fed to the third reactor where dehydrogenation and cracking take place. The design octane number for reformate of this process is 95–108. In CCR, the catalysts are regenerated continuously and have to be more resistant and tolerate high coke level than the catalysts used in SRR giving higher selectivity to aromatics [7]. The efficiency of the reforming process is related to the catalyst performance, and many factors like coke formation, contamination on the active site, catalyst agglomeration and poisoning might decrease and cause catalyst deactivation which affects its performance. Activity could be restored by regeneration and after that the catalyst will retain its high surface area, dispersion of the metal should be high with proper acidity level [4].

The focus on naphtha catalytic reforming is rare and most of the past study deals with reforming in batch reactors using pure synthetic fuel [8] on contrary to steam and carbon dioxide reforming which are widely studied. In the present study, bi-functional zeolite was introduced for the first time for the catalytic reforming reaction of heavy naphtha in a packed bed continuous reactor. Nano zeolite HY was prepared and loaded with Pt and Ti and packed in the reformer for naphtha upgrading. Also, the performance of commercial HY zeolite (with the same Pt–Ti loading) and Pt–Re/Al_2O_3 was also investigated in the same pilot plant for octane number comparison.

Experimental section

Feed Stock

Heavy Naphtha is supplied by the Midland Refineries Company/Al-DURA refinery as raw material for reforming activity test. Some properties of Heavy Naphtha as given by the supplier are listed in Table 1.

Catalysts

Five samples of the bi functional catalysts was prepared and loaded with different percentages of platinum and titanium on the prepared nano zeolite HY (71 nm) and

Table 1 Properties of Heavy Naphtha

Properties	
Sulfur content (ppm)	1.3
Density (g/cm^3)	0.74
Chemical composition (wt%)	
Paraffins	71
Naphthenes	11
Aromatics	18
RON	57

on commercial HY for comparison. Details of preparation and catalyst characterization can be found elsewhere [9]. The physical and chemical properties of catalysts are given in Table 2. Commercial Pt–Re/Al$_2$O$_3$ is supplied from Al-Basrah Refinery.

Catalyst performance

Catalytic activity of the samples of the prepared Pt–Ti/HY zeolite was carried out in the reforming pilot unit which was supplied by VINCI Technology. Images of the equipment with the individual parts can be found in the supplementary information files.

The unit consists of a fixed bed carbon steel reactor (1.5 m length, 2.5 cm O.D.) equipped with control and cooling systems. Each experiment was carried out by charging the reactor with 60 g of the catalyst sample while the upper and lower parts of the reactor were filled with ceramic balls (5–6 mm). Three reaction temperatures of 490, 500, 510 °C under operating pressure of 10 bars and H$_2$/Hydrocarbon ratio of 3 and 1.4 h^{-1} LHSV were investigated. Before the performance experiment the catalyst was reduced with hydrogen gas at 450 °C for 3 h.

Heavy naphtha was pumped and mixed with hydrogen then passed through catalyst bed, and the reaction takes place. The product was cooled by the cooling

Table 2 Properties of the Prepared Catalysts [9]

Catalyst type	Surface area (m^2/g)	Pore volume (cm^3/g)	Bulk density (g/cm^3)	Crushing strength (kg)
(0.25 wt% Pt, 2.2 wt% Ti)/HY	337	0.213	0.671	1.76
(0.13 wt% Pt, 1 wt% Ti)/HY	357	0.38	0.701	1.94
(0.25 wt% Pt, 1 wt% Ti)/HY	402	0.209	0.649	0.65
(0.13 wt% Pt, 0.75 wt% Ti)/HY	550	0.392	0.695	1.25
(0.13 wt% Pt, 0.75 wt% Ti)/commercial HY**	575	0.381	0.601	0.83
(0.3 wt% Pt, 0.3 wt% Re/Al$_2$O$_3$)*	220	0.6	0.57	–

*Al-Basrah refinery catalyst, **loaded on commercial zeolite

system and separated in a separator to produce the "reformate" which was collected and analyzed using GC analysis(Type, 3300 Varian) for Paraffin, Aromatic and Naphthenes, also Research Octane number " RON" was measured using portable octane no. analyzer. After the experiment carbon content, abrasion percent and crushing strength for the catalyst were investigated for the stability assessment and the mechanical resistance of the prepared catalysts through the process.

Results and discussions

Effect of operating conditions on catalytic reforming

It can be seen that the octane number of the feed of heavy naphtha was enhanced using all samples of the catalyst with different extents at the whole range of temperature studied. The maximum increase was 27 units at the process operating conditions of 10 bars and 510 °C. The quality of the produced reformate can be represented by octane number and the results showed that research octane number ranged from 77 to 84 as shown in Tables 4, 5, 6, 7, and 8. The sample of catalyst with 0.13 wt% Pt and 0.75 Ti wt% loading gave the highest octane number of 84. This can be attributed to the metallic function of the acidic catalyst and also, this sample possesses the highest surface area among others. This loading was found to increase the dehydrogenation of Naphthenes and the dehydro-cyclization of Paraffins leading to the increase in the aromatic content which in turn is the reason behind the octane number enhancement of the reformate [10, 11]. The results of the same loading of 0.13% Pt and 0.75% Ti which was applied to the commercial HY catalyst for comparison, showed a good agreement at the range of the temperature studied at 10 bars with octane number of 86.2. This gives the indication that the reforming reaction is favored by the effective metal loading as well as by high surface area of the support which lead to the desired increase of octane number of the produced naphtha. On the same time, a slight increase of catalyst coking is noticed as shown in Table 3 where the carbon content was increased ranging from 7 to 9%. The build up of carbonaceous materials can be reduced and controlled by using hydrogen gas promoting long catalyst life. However, hydrocracking is an undesirable reaction since it produces gaseous product but it may contribute to

Table 3 Carbon content of the prepared catalysts

Catalyst type	Carbon content (wt%)
(0.25 wt% Pt, 2.2 wt% Ti)/HY	9.11
(0.13 wt% Pt, 1 wt% Ti)/HY	8.25
(0.25 wt% Pt, 1 wt% Ti)/HY	7.56
(0.13 wt% Pt, 0.75 wt% Ti)/HY	9.13
(0.13 wt% Pt, 0.75 wt% Ti)/commercial HY	8.08

Table 4 Octane number versus reaction temperature at 10 bar using 0.25 wt% Pt, 2.2 wt% Ti/HY, H_2/HC ratio = 3, LHSV = 1.4 h^{-1}

Temperature (°C)	RON
490	78
500	79
510	77

RON of Naphtha feed = 57

Table 5 Octane number versus reaction temperature at 10 bar using 0.13 wt% Pt, 1 wt% Ti/HY, H_2/HC ratio = 3, LHSV = 1.4 h^{-1}

Temperature (°C)	RON
490	79
500	79
510	80

RON of Naphtha feed = 57

Table 6 Octane number versus reaction temperature at 10 bar using 0.25 wt% Pt, 1 wt% Ti/HY, H_2/HC ratio = 3, LHSV = 1.4 h^{-1}

Temperature (°C)	RON
490	81
500	77
510	77

RON of naphtha feed = 57

Table 7 Octane number versus reaction temperature at 10 bar using 0.13 wt% Pt, 0.75 wt% Ti/HY, H_2/HC ratio = 3, LHSV = 1.4 h^{-1}

Temperature (°C)	RON
490	80
500	81
510	84

RON of Naphtha feed = 57

Table 8 Octane number versus reaction temperature at 10 bar using 0.13 wt% Pt, 0.75 wt% Ti/commercial HY, H_2/HC ratio = 3, LHSV = 1.4 h^{-1}

Temperature (°C)	RON
490	86.6
500	86.2
510	80.4

RON of Naphtha feed = 57

octane enhancement by cracking of large molecular like n-decane to C_3 and C_7 and hence, the latter can be transformed to aromatic through aromatization reaction [12].

By comparing the behavior under the present applied pressure (10 bar) and the pressure of 20 bar which was studied previously [13], it can be reported that the

octane number using 20 bar reached 86 achieving two points increase than the one obtained in the present work at 10 bars however, raising the operating pressure giving an increase in hydro cracking reactions which leads to reduce the yield of the produced reformate but decreases the coking of catalyst leading to longer cycle life. This conclusion was reported earlier where lower yield of reformate leads to higher octane number meaning one output on the expense of the other due to their inverse relationship [11, 14]. The maximum increase in the research octane number using the sample 0.13 Pt and 0.75 Ti/HY zeolite was 40.35, 42.11 and 45.6% and 43.86, 47, 37 and 50.88% at the studied range of temperature and 10 and 20 bars respectively. This is shown in Table 9.

To show the validity of the prepared catalyst for naphtha reforming reaction, the conventional catalyst of Al-Basrah Refinery has been tested at the same reforming unit at 510 °C and 20 bars. The octane number obtained was 88 for the conventional catalyst compared with 86 and 86.6 for the prepared catalyst as shown in Table 10. It can be deduced that these slight differences in the results can be overcome and the metallic loading had the pronounced effect in the octane number enhancement compared to the differences in the support surface area.

Catalyst performance and reformate yield

Generally the activity of a catalyst means how well the performance with respect to the reaction rate is which means that the higher is the reaction rate, the higher is the content of the desired product and specifically in the catalytic reforming higher yield of aromatic. The acidic properties of the support are influenced by the metal loading and this leads to affect the activity and selectivity of the prepared catalysts. Reformate yield is defined as the volume of product reformate to the volume of heavy naphtha feed. Total yield of reformate depends strongly on the total conversion of naphtha and as the quality of reformate increases, the yield decreases and vice versa [15, 16]. The results showed that the reformate yield ranges from 89 to 94% for all samples of the catalyst investigated as shown in Fig. 1 and the sample of producing high octane number had low reformate yield as abovementioned.

The lowest metal loading of 0.13% Pt and 0.75% Ti showed a faster rate of deactivation as indicated by its higher carbon content than other samples, even though it is within the acceptable limits. It can be related to its higher activity towards hydrogenation–dehydrogenation reactions and its contribution to naphtha upgrading.

Table 9 Octane number versus reaction temperature at 10 and 20 bar using 0.13 wt% Pt, 0.75 wt% Ti/HY, H_2/HC ratio = 3, LHSV = 1.4 h^{-1}
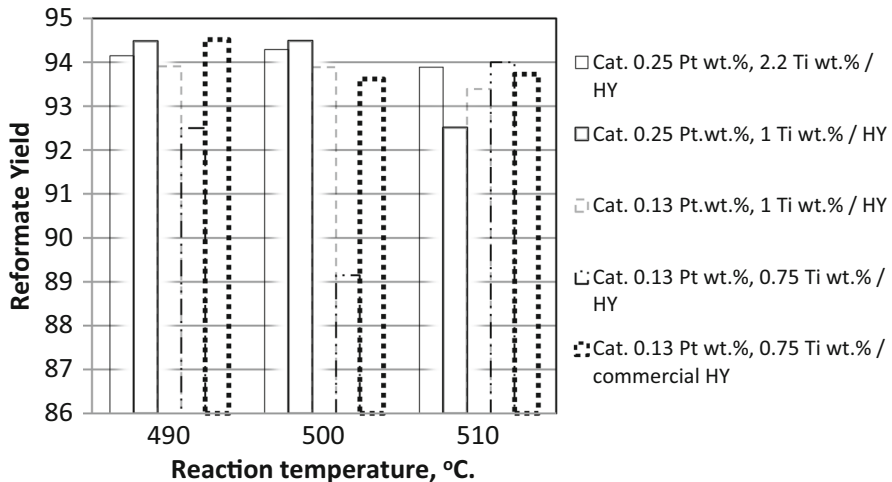
Temperature (°C)	RON at 10 bar	RON at 20 bar
490	80	82
500	81	84
510	84	86

RON of Naphtha feed = 57

Table 10 Comparison of octane number using different catalysts

Catalyst	Reaction temperature (°C)	Pressure (bar)	RON
(0.3 Pt wt%, 0.4 Re wt%)/Al$_2$O$_3$	510	20	88
(0.13 Pt wt%, 0.75 Ti wt%)/HY	510	20	86
(0.13 Pt wt%, 0.75 Ti wt%)/HY**	510	20	86.6

*Al-Basrah refinery catalyst, **loaded on commercial zeolite

Fig. 1 Reformate yield for all samples of the catalysts versus the reaction temperature and 10 bars. H$_2$/HC = 3, LHSV = 1.4 hr^{-1}

Effect of bimetallic loading on catalytic reforming

Although HY zeolite catalyst exhibits hydrophobic properties derived from its high SiO$_2$/Al$_2$O$_3$ ratio of 5, the TiO$_2$ loading on Pt/HY seems to give a higher hydrophobicity leading to achieve extra efficient activity. Fig. 2 shows the incorporation of TiO$_2$ in the frame work structure of the Zeolite, which was proposed by [17]. Titanium bounds directly to the surface of zeolite and during calcinations, TiO^{+2} reacts with lattice oxygen to give TiO$_2$ or TiO$^+$ in the framework through Ti–O–Si bonds forming strong electrostatic fields with polar adsorbate species giving good catalytic activity of zeolite [17, 18]. TiO$_2$ contains isolated and tetrahedrally coordinate titanium oxide species in the frame work of zeolite giving more stable catalyst and much higher selectivity towards isomerization and aromatization reactions because Ti modified zeolite enhances the hydrogenation–dehydrogenation metallic function of Pt active site also balancing the total acidity of HY zeolite resulting in an efficient balance between metallic and acidic function of catalyst [18–20].
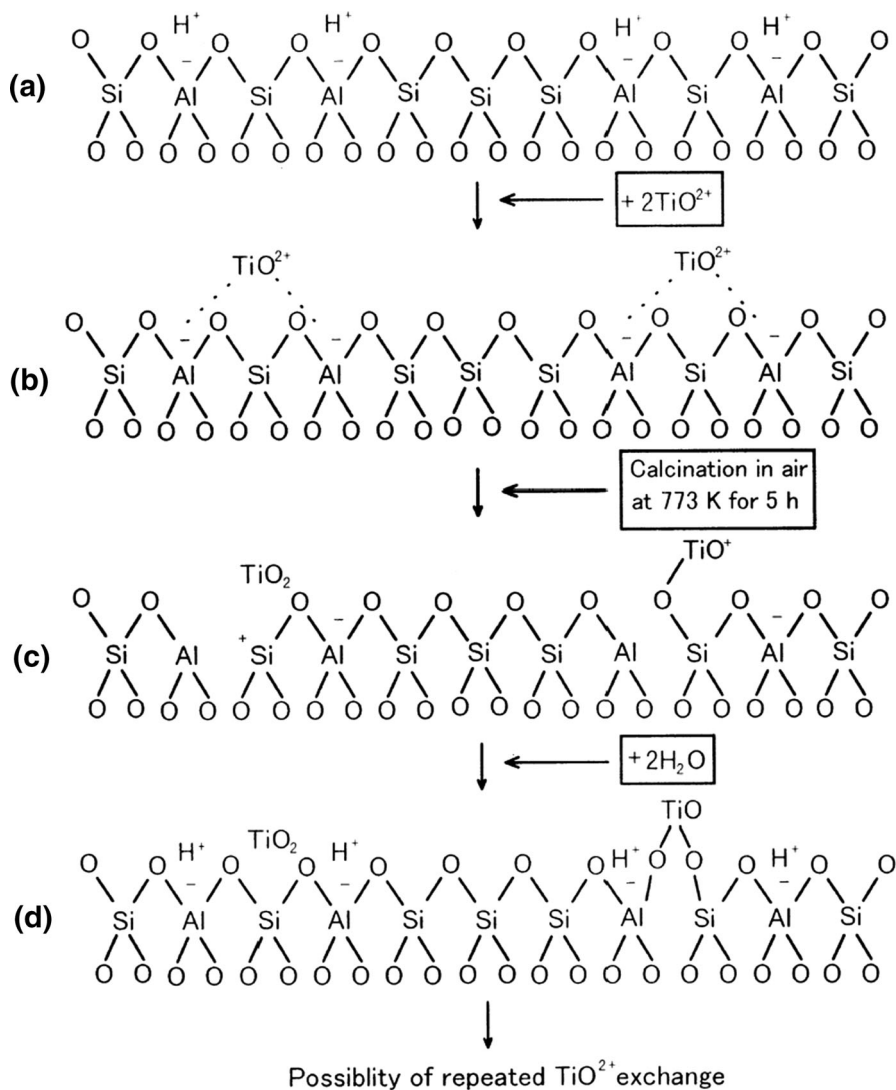
Fig. 2 TiO_2 incorporation into the framework structure of zeolite [18]

Another advantage of the bimetallic loading in the composite catalyst could be related to that Titanium may form an alloy with platinum preventing the increase in platinum crystallites size and keeping it well dispersed (prevent sintering) also preventing coke precursors to deposit onto the surface of catalyst [12]. The acidic properties of the support are influenced by the metal loading and this leads to affect the activity and selectivity of the prepared catalysts.

Catalytic naphtha reforming reactions are reported to proceed typically on both metallic sites represented by Pt & Ti on the surface of the bi-functional catalyst in addition to the acidic site represented by HY zeolite. Fig. 3 shows a possible
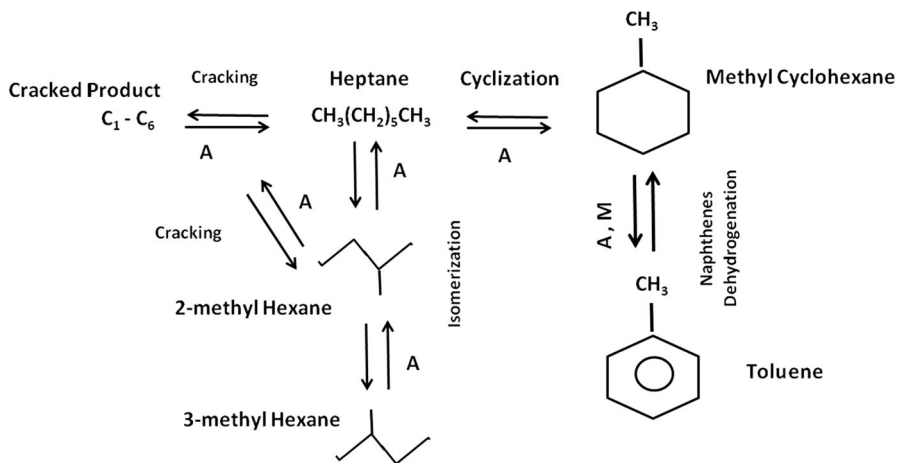
Fig. 3 Proposed Mechanism of the Reforming reaction on Metallic (M) and Acidic (A) Sites of Pt–Ti/HY Bi-functional zeolite

mechanism for a typical hydrocarbon of seven carbon atoms as suggested for Heptane. This figure demonstrates that some reactions are promoted by metallic loading like hydrogenation–dehydrogenation reactions while catalytic cracking and cyclization reactions are promoted by both metallic and acidic properties of the catalyst. At all cases, it can be deduced that the metal loading got the important role in all catalytic reforming reactions.

Octane number correlation

The reformate quality depends on its composition and types of hydrocarbons present. Octane number characterizes the quality of reformate and this is referred to clean octane number because it is calculated or measured without any additives that are used to enhance the octane number [21]. In an attempt to find a correlation relating composition to octane number, a correlation was found for calculating the octane number in terms of the boiling point for each hydrocarbon family present in the reformate product i.e., Paraffin, Naphthene and Aromatic and is given by Eq. 1 [22].

$$RON = 31.0621 + 0.53542 * T \tag{1}$$

Where, T is the boiling point in °C for the individual component. Then the octane number of reformate is calculated using this equation:

$$RON = XP * (RON)P + XN * (RON)N + XA * (RON)A \tag{2}$$

Where, XP, XN and XA are the volume fractions of Paraffin, Naphthene and Aromatic compounds [22] respectively. Comparison between the measured and the calculated results is listed in Table 11 for the sample of 0.13% Pt–0.75% Ti/HY. The absolute error was 5.3% at the studied operating temperatures and pressures.

Table 11 Comparison between measured and calculated results of octane number for (0.13 Pt wt% –0.75 Ti wt%)/HY catalyst

Reaction temperature (°C)	Pressure (10 bar)		Absolute Error (%)
	RON (Measured)	RON (Calculated)	
490	80	80.008	0.01
500	81	80.076	1.14
510	84	80.1	3.9
Reaction temperature (°C)	Pressure (20 bar)		Absolute Error (%)
	RON (Measured)	RON (Calculated)	
490	82	83.53	1.8
500	84	81.65	2.7
510	86	81.38	5.3

Conclusions

From the present study it can be concluded that loading titanium on Pt/HY zeolite had a pronounced good catalytic activity toward reforming of heavy naphtha. Samples of the aforementioned catalyst with different loading percentages had a perfect role in the enhancement of octane number and yield of reformate which is the product of processing heavy naphtha feed. Pilot plant encouraging results can be the future candidates for the refinery catalyst. The most interesting property of the present catalyst is its low platinum loading (0.13) compared to the corresponding loading of the commercial Al_2O_3 catalyst (0.3). This would be an advantage from the economic point of view due to the fruitful decrease in loading. The big role of the present metal is its contribution in the hydrogenation–dehydrogenation reactions in addition to the appropriate zeolite support chemical and mechanical properties. This work gives Zeolites the opportunity to replace past conventional catalytic reforming catalysts. It's worth noting that there is no need to chlorine loading and/or replenish as in the commercial type.

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Affiliations

Nada S. Ahmedzeki[1] · Ban A. Al-Tabbakh[2] · Maher B. Antwan[2] · Selahattin Yilmaz[3]

Ban A. Al-Tabbakh
dr.banaltabbakh@gmail.com

Selahattin Yilmaz
selahattinyilmaz@iyte.ed.tr

[1]   Chemical Engineering Department, College of Engineering, University of Baghdad, Baghdad, Iraq

[2]   Petroleum Research & Development Center, Ministry of Oil, Baghdad, Iraq

[3]   Department of Chemical Engineering, Izmir Institute of Technology, Izmir, Turkey