



Enhancement of Iraqi Light Naphtha Octane Number Using Pt Supported HMOR Zeolite Catalyst

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Abstract

The hydroconversion of Iraqi light straight run naphtha was studied on zeolite catalyst. 0.3wt.%Pt/HMOR catalyst was prepared locally and used in the present work. The hydroconversion performed on a continuous fixed-bed laboratory reaction unit. Experiments were performed in the temperature range of 200 to 350°C, pressure range of 3 to 15 bars, LHSV range of 0.5-2.5h⁻¹, and the hydrogen to naphtha ratio of 300.

The results show that the hydroconversion of Iraqi light straight naphtha increases with increase in reaction temperature and decreases with increase in LHSV.

High octane number isomers were formed at low temperature of 240°C. The selectivity of hydroisomerization improved by increasing reaction pressure up to 15 bars. Catalyst activity almost stable and independent of time on stream at 15 bar up to 20 h.

Keywords: Hydroisomerization, Light naphtha, Zeolite, Octane number, Mordenite.

1. Introduction

With the continuously increasing environmental concerns and stringent regulations on the utilization of gasoline as an automotive fuel, there is a great need to search for alternative gasoline octane number enhancers. Currently, various additives are available for gasoline octane number enhancement e.g. oxygenate and aromatic compounds (Alexander et al., 2013; Sandeep et al., 2013; Liu et al., 2011; Nikolaou et al., 2004; Chao et al., 1996). These compounds are considered as environment harmful compounds. Finding a harmless substituent to the high-octane oxygenates and aromatics is not a straightforward task, since main octane enhancers have either been completely phased out due to their toxicity in many countries worldwide, such as in the case

of lead-containing additives, or seen their use decline due to their environmental problems, like oxygenates such as MTBE (Methyl t-butyl ether). In addition, using octane-rich reformates fails to do the trick since aromatics are a major constituent of them (Busto et al., 2012; Chiang et al., 2011; Chao et al., 2000). However, the utilization of an upgraded low-value refinery stream to the gasoline pool might present a solution to the problem, as it can considerably lower the cost of gasoline production, while losing only some of its original quality, but still remain environmentally friendly.

It is a promising objective to produce high octane number gasoline from petroleum sources. The requirements to reduce these additives in

gasoline present a need to find an alternative way to maintain its research octane number (RON). An alternative way is to increase the RON for the paraffinic content of gasoline, which can be accomplished through hydroisomerization. Light paraffin hydroisomerization has been used provide a cost-effective solution to manage benzene in motor fuels (Stanislav et al., 2012; Raed et al., 2010; Liu et al., 2009; Theo et al., 2008).

The process of light naphtha hydroisomerization involves the transformation of normal paraffins (i.e. normal C₅-C₇ fraction which is the main constituent) into their isomers, which have higher octane numbers than the normal paraffins over acidic catalysts (Sege et al., 2003; Gauw et al., 2002; Lee et al., 1997; Guisnet et al., 1991).

Mechanically, hydroisomerization occurs in three elementary steps. First, the alkane is dehydrogenated. Then generated alkene adsorbs on a Brønsted acid site, forming an alkoxy group (a carbenium ion in the transition state), which isomerizes and eventually desorbs. Finally, the iso-olefin is hydrogenated to iso-alkane. Therefore, catalysts are bifunctional, with a metal (Pt, Pd) catalyzing the hydrogenation/dehydrogenation step and an acidic function for the formation and isomerization of the alkoxy group/ carbenium ion. The metal component also helps reduce catalyst deactivation by hydrogenation coke precursors (Gauw et al., 2002; Hollo et al., 2002; Aboul-Gheit et al., 1998; Allian et al., 1997).

As the reaction proceeds via carbenium ions, other Brønsted acid catalyzed reactions, as oligomerization and cracking compete with isomerization (Gauw et al., 2002; Hollo et al., 2002). In addition, side reactions on the metal, as hydrogenolysis of the alkane to smaller alkanes, may reduce the selectivity of the overall hydroisomerization reaction (Gauw et al., 2002; Hollo et al., 2002).

In the present study, Pt supported HMOR zeolite catalyst is prepared locally and used to hydroisomerize Iraqi light straight run naphtha (LSRN).

The main objective in this work is to develop a useful catalyst and optimize the limited ranges of operating conditions (temperature, LHSV, and operating pressure) on the naphtha transformation and octane number in a fixed bed continuous laboratory reaction unit. Catalyst stability is also highlighted.

2. Experimental Work

2.1. Materials

2.1.1. Feedstock

Light straight run naphtha (LSRN) supplied from Al-Dura Refinery was used as a feedstock in hydroisomerization experiments. The properties of LSRN are listed in Table. 1

Table 1,
Properties of straight run light naphtha.

API @ 60°/60°F	78.6
Octane Number	61.4
Sulfur Content	< 2ppm
Kinematic Viscosity at 25°C	7.2×10 ⁻⁷ m ² /s
Composition	Wt.%
Propane	0.01
i-butane	3.19
n-butane	4.27
i-pentane	33.46
n-pentane	21.57
cyclo-pentane	0.94
i-hexane	6
n-hexane	15.27
cyclo-hexane	4.1
i-heptane	2.5
n-heptane	4.58
i- and n-octane	1.37
Benzene	0.76
Toluene	0.95
Xylene	0.16
Naphthalene C ₈	0.2
Naphthene C ₈	0.39
Naphthene C ₉	0.28

2.1.2. Hydrogen and Nitrogen Gases

High purity (99.999 vol. %) of hydrogen and nitrogen gases supplied from the local market were used in the present work.

2.1.3. Ammonium Mordenite Zeolite

Ammonium mordenite zeolite (CBV-21A) supplied as a powder from Zeolyst International Company. It was used in the preparation of the proposed catalyst. The properties of this zeolite are listed in Table 2.

Table 2,
Properties of ammonium mordenite zeolite powder (CBV-21A).

SiO ₂ /Al ₂ O ₃ Mole Ratio	Nominal Cation Form	Na ₂ O Weight %	Surface Area, m ² /g
20	Ammonium	0.08	500

2.1.4. Hexachloroplatinic Acid

Hexachloroplatinic acid (H₂PtClO₆.6H₂O) of an analytical grad (40 wt.% Pt) was supplied by Fluka Chemi AG.

2.2. Catalyst Preparation

100 g of Ammonium mordenite (CBV-21A) powder was shaped as a pellets with 3mm×5mm using a laboratory scale pelleting machine (model TDP-1.5 from MINHUA PHARMACEUTICAL MACHINERY CO., LIMITED). The final form was dried at 110°C and stored in an evacuated place.

2.3. 0.3 wt.%Pt/ HMOR Zeolite Preparation

0.3wt%Pt on HMOR zeolite was prepared by impregnation method. HMOR pellets were dried over night at 110°C and impregnated with 0.3 g of H₂PtClO₆.6H₂O in 10 ml deionized water. The final impregnated pellets were dried at 110°C over night and calcinated at 300°C for 3 hrs in a furnace with dry air. The calcinated catalyst pellets were then reduced with hydrogen at 350°C

for 3 hrs (Al-Hassani, 2007). The properties of the prepared catalyst are listed in Table 3.

Table 3,
Properties of the 0.3wt%Pt/HMOR zeolite.

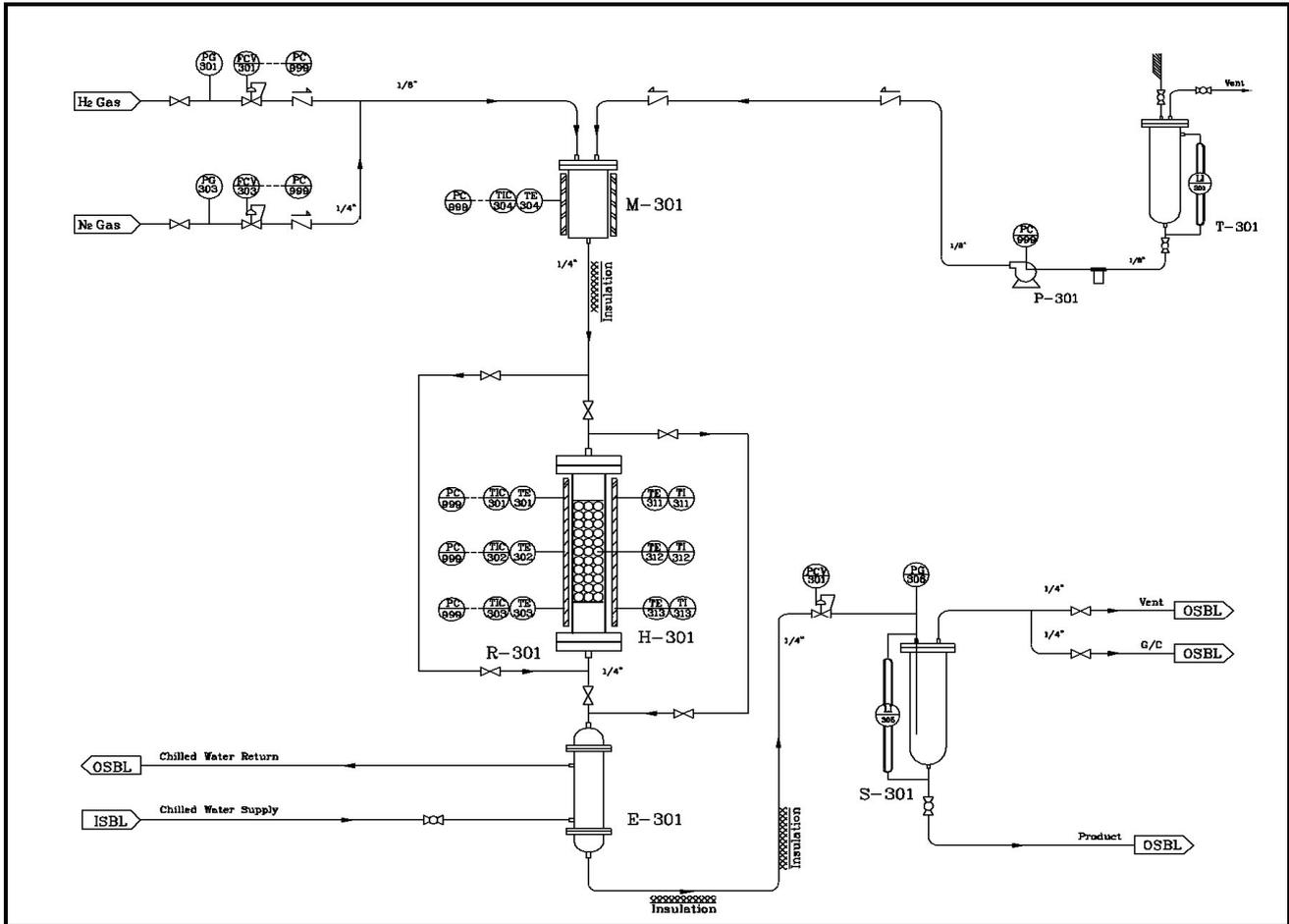
Surface Area m ² /g	662
Bulk density g/cm ³	0.592
Pore volume, cm ³ /g	0.143

2.4. Hydroisomerization Reaction Unit

Hydroisomerization experiments were conducted in a continuous fixed bed reactor laboratory scale unit. Figure 1 shows the schematic diagram of this unit. It consist of feed tank (T-301), gas flow meter and controller (FCV), feed pump (P-301), evaporator (M-301), fixed bed reactor (R-301), high pressure separator (E-301), low pressure separator (S-301), and an appropriate heating system (H-301). The reactor was heated and controlled automatically with computer control software and by four steel-jacket heaters using chromal alumel thermocouple (type k).

2.5. Hydroisomerization Experiments

30 cm³ of fresh catalyst was charged to the reactor and between two layers of inert material (glass balls). In the beginning of each experiment, the reactor was flashed with nitrogen 2 l/h for 1 h to purge the air from the system, then the reactor is heated to the desired temperature. When reactor temperature is reached, the nitrogen valve is closed. A pre-specified flow rate of light naphtha was set on, vaporized in the evaporator and the vapor was mixed with the hydrogen in the mixing unit at a specified flow rates. The mixture entered the reactor from the top, distributed uniformly and reacted on the catalyst. The gaseous products passed through the high pressure separator and the final condensates were collected in the low pressure separator only after steady state operation was established and the initial products were discarded. The hydroisomerization reaction conditions employed are temperature range 200-350°C, liquid hourly space velocity (LHVS) range 0.5-2.5h⁻¹, hydrogen to light naphtha volumetric ratio 300 and the pressure was ranging from 3-15bar.



R-301	Tubular Reactor	P-301	Feed Pump	FCV-301 FCV-302 FCV-303 FCV-304	Mass flow controller
T-301	Feed Tank	E-301	Condenser		
S-301	Separator	M-301	Mixer		
H-301	Heater	PRV-301	Pressure Relief Valve		
Piping and Instrument Flow Diagram					
Laboratory Reaction Unit					
University of Baghdad					
Al-Khwarizmi College of Engineering					
Department of Biochemical Engineering					

Fig. 1. Schematic Diagram of Laboratory Continuous Fixed Bed Reaction Unit.

2.6. Analysis

2.6.1. Atomic Absorption Analysis

The analysis of platinum, in the prepared catalyst, was achieved using atomic absorption spectrophotometer (model PYE-UNICAM SP9) in IBN Sina State Company.

2.6.2. Gas Chromatographic Analysis

The collected products (liquid and gas) were analysis into their components by gas chromatographic (GC) analysis using SHIMADZU GC model 2014A with FID detector.

2.6.3. Octane Number Test

This test was achieved in DURA refinery using RON method (ASTM -D 2699).

3. Results and Discussion

The results of hydroconversion runs of Iraqi LSRN are discussed in this section in order to evaluate the catalyst performance. The purpose of the present work is to choose the optimal experimental conditions that most satisfy high catalyst stability and product octane number. The hydroconversion involve three main reactions hydroisomerization, hydrocracking, and hydrocycalization and aromatization. In this section, discussions were built upon the results of these reactions.

3.1. Effect of Temperature

The effect of temperature on LSRN transformation is shown in Figs. 2-4. It is clearly temperature dependent; in fact, in the temperature range of 473 to 513K the hydroconversion shifted towards the hydroisomerization conversion and as shown in Fig. 3, while above 513 the isomers formation decreases and the reaction is shifted towards the hydrocracking, hydrocycalization aromatization reactions and as shown in Figs. 2 to 4. This means that the formation of isomers is favored at lower temperature and this phenomenon is further explained by Fig. 5. The octane number of n-hexane is around 25, n-pentane is around 62, the MP isomer is around 75 and DMB is around 95, furthermore, the lower reaction temperature, the greater the percentage of branched alkane at thermodynamic equilibrium,

hence the higher the octane number (Sege, 2003; Gauw et al., 2002; Grillo et al., 1997).

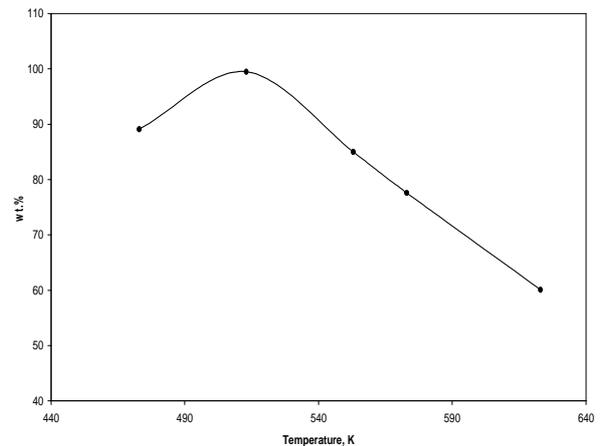


Fig. 2. Isomers Formation Percent vs. Temperature at LHSV of 0.5h^{-1} and Pressure of 15bar.

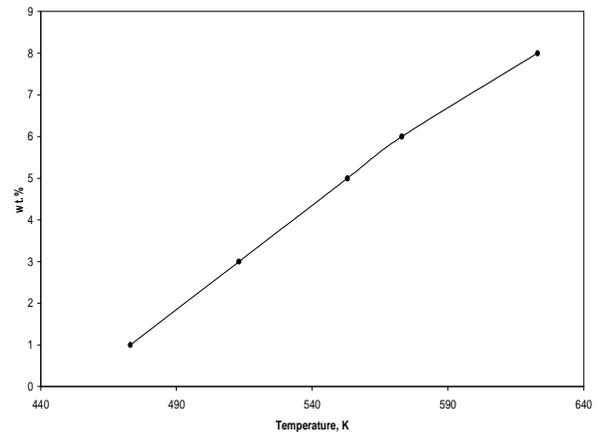


Fig. 3. Hydrocracking Products Formation Percent vs. Temperature at LHSV of 0.5h^{-1} and Pressure of 15bar.

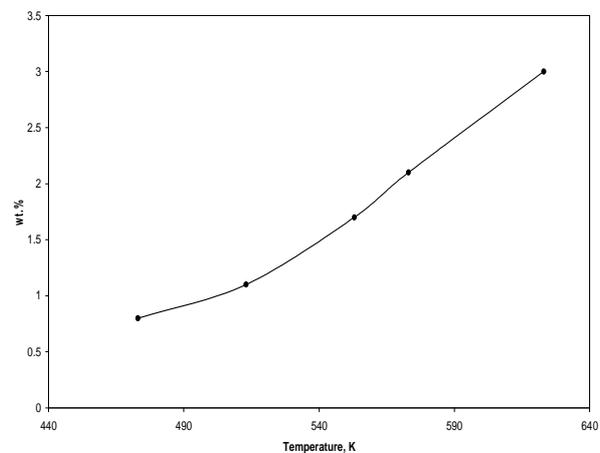


Fig. 4. Percent of Hydrocyclization and Aromatization Products vs. Temperature at LHSV of 0.5h^{-1} and Pressure of 15bar.

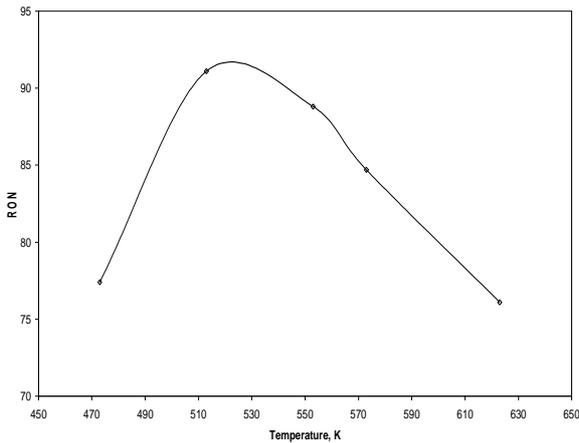


Fig. 5. Effect of Temperature on the RON at LHSV of 0.5 h⁻¹ and Pressure of 15bar.

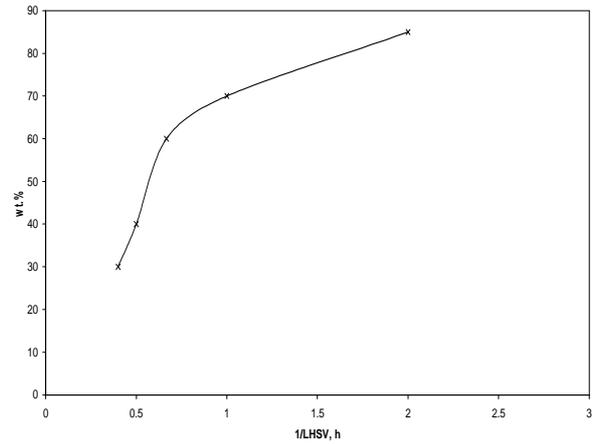


Fig. 6. Isomers Formation Percent vs. LHSV at Temperature of 553K and Pressure of 15bar.

3.2. Effect of Contact Time

Figures 6 to 8 show the change in LSRN transformation as a function of contact time. The contact time is expressed as the reverse of LHSV, the liquid hourly space velocity taken as the ratio between the volumetric flow rate of LSRN and catalyst volume. It can be observed from these figures that as the contact time increases the hydroconversion increases so the rates of hydroisomerization, cracking and hydrocyclozation and aromatization increases. Also isomers are formed at low contact times (i.e. low conversion), a small amount of cracking products appearing at higher contact times.

A high increase in RON number was observed with the increase in contact time and as shown in Fig. 9. This increases many be attributed to the formation of high octane number isomers and as explained above. The same observations were reported in other works (Busto et al., 2012; Jiménez et al., 2003; Partylak et al., 1998; Allian et al., 1997).

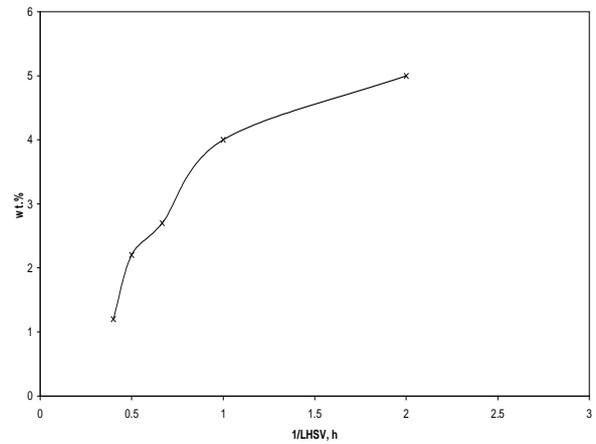


Fig. 7. Hydrocracking Products Formation Percent vs. LHSV at Temperature of 553K and Pressure of 15bar.

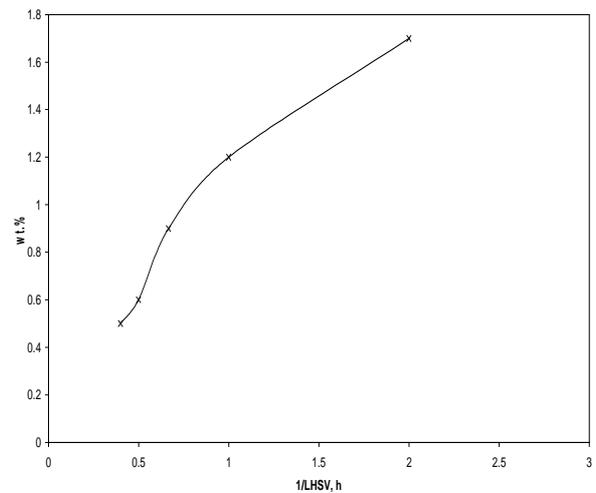


Fig. 8. Percent of Hydrocyclozation and Aromatization Products vs. LHSV at Temperature of 553K and Pressure of 15bar.

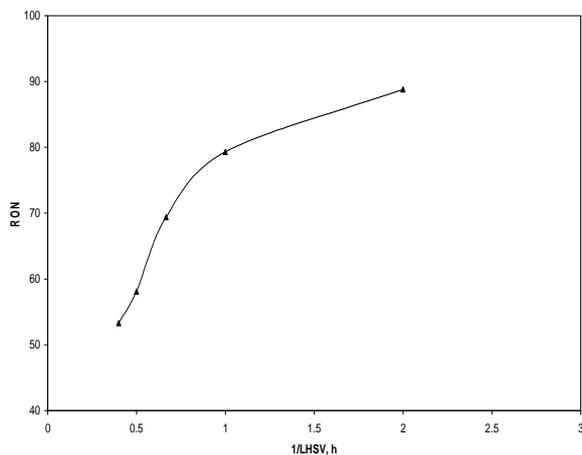


Fig. 9. Effect of Contact Time on RON at Temperature of 553K and Pressure of 15bar.

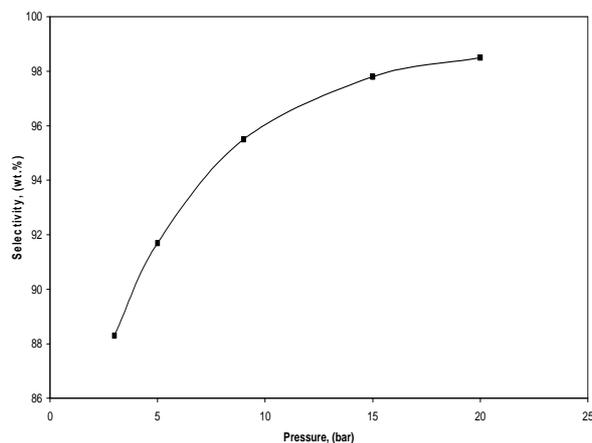


Fig. 10. Hydroisomerization Selectivity vs. Operating Pressure at LHSV of 1h-1 and Temperature of 553K.

3.3. Effect of Pressure

The effect of pressure on the Iraqi LSRN reaction selectivities and RON are shown in Figs. 10, 11, and 12. As the operating pressure increases the selectivity of hydroisomerization increases and the selectivity of hydrocracking decreases rapidly while the selectivity of hydrocyclization and aromatization slightly increases. The increase in operating pressure is probably leads to a decrease in alkenes formation, in turn, diminish the probability to proceeds the dimerization cracking and provide higher isomerization selectivity as shown in Fig. 10. As a results RON increases rapidly with the increases in the operating pressure and up to 15bar, after this value RON unaffected by the change in operating pressure. The reaction of alkane transformation proceeds with the skeletal hydroisomerization via bimolecular mechanism. Abuda Wood et al.; 2010, showed that the hydrogenation activity increased at a higher hydrogen pressure that results in the hydrogenation of more intermediate olefins, which can be due to a shorter intermediate olefin residence time inside the catalyst, and thus minimizing the cracking activity. These observations are agree well with the results reported by Gauw et al., (2002), Liu et al., (1997), Chao et al., (1996) and Guisnet et al., (1991); Chao et al., 2000.

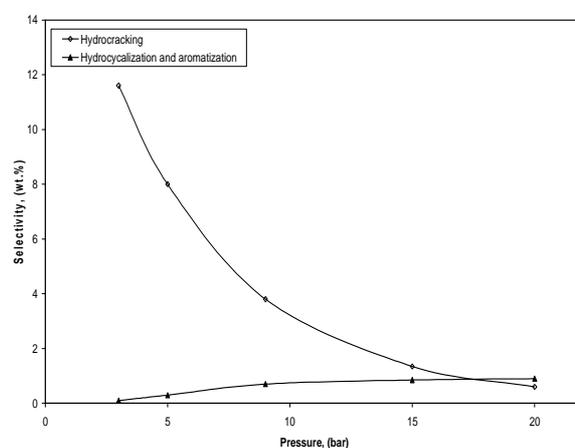


Fig. 11. Hydrocracking, Hydrocyclization and Aromatization Selectivities vs. Operating Pressure at LHSV of 1h-1 and Temperature of 553K.

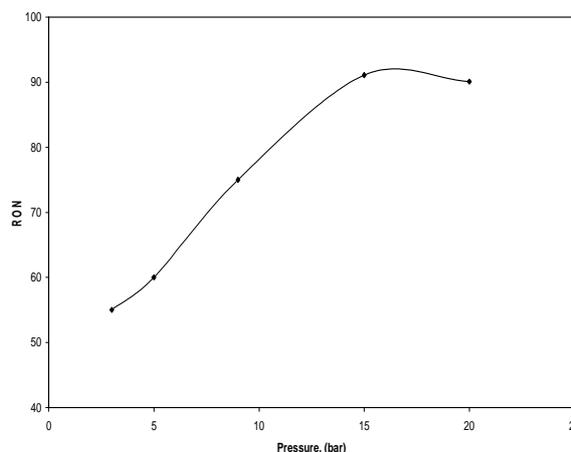


Fig. 12. Effect of Operating Pressure on RON at LHSV of 1h⁻¹ and Temperature of 553K.

3.4. Catalyst Stability and Time on Stream

The stability during the reaction of zeolite catalysts is measured by Time on stream. It can be observed from Fig. 13 that the present 0.3wt.%Pt/HMOR catalyst is very stable with almost no (i.e. very slight change) change in activity when tested for 20 hours. Unfortunately, this stability is in the expense of hydroisomerization activity towards high octane numbers isomers. This is partly due to a very slow coking that may have poisoned the acid sites on the catalyst and changed the effective amount of acid sites density on the catalyst needed. These results are agree well with other observation reported by Stanislav et al., 2012; Theo et al., 2008; Nikolaou et al., 2004; Gauw et al., 2002; Burckle et al., 2000; Chao et al. 2000.

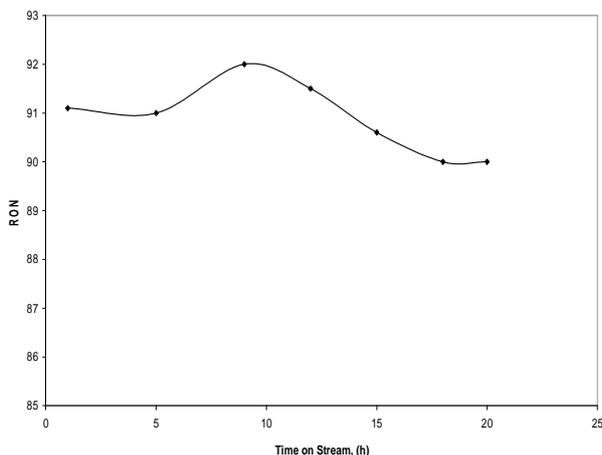


Fig. 13. Effect of Time on Stream on RON at LHSV of 1h-1 and Temperature of 553K.

4. Conclusions

The hydro transformation of Iraqi LSRN was evaluated in a laboratory fixed bed reaction unit using 0.3wt.%Pt/HMOR zeolite catalyst. The following conclusions were drawn from the experimental results:

1. The prepared catalyst exhibits a high hydroisomerization activity within the studied range of operating conditions.
2. The hydroisomerization reaction is temperature dependent, and the lower temperature the greater hydroisomerization selectivity and in turn high RON value. The optimum reaction temperature ranging

between 240-280 for producing high octane number isomers.

3. The selectivity of LSRN hydroisomerization on 0.3wt.%Pt/HMOR catalyst could be significantly improved by increasing reaction pressure and reducing the reaction temperature. The increase of reaction pressure also causes decrease in the cracking selectivity on the catalyst.
4. The activity of 0.3wt.%Pt/HMOR catalyst almost independent of time on stream at 15 bar up to 20h.

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Nomenclature

HMOR	H-Mordenite
LHSV	Liquid hourly space velocity
LSRN	Light straight run naphtha
RON	Research octane number
MP	Methyl Pentane isomer
DMB	Dimethyl butane isomer

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تحسين الرقم الاوكتاني للنافثا العراقية الخفيفة باستعمال العامل المساعد Pt المحمل على الزيولايت HMOR

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الخلاصة

تمت دراسة تحول النافثا العراقية باستعمال الزيولايت كعامل مساعد. تم تحضير العامل المساعد 0.3wt.%Pt/HMOR مختبريا واستعماله في البحث الحالي. اجريت تجارب التحول باستعمال وحدة تفاعل مختبرية من نوع الحشوة الثابتة. اجريت التجارب بمدى من درجات الحرارة تراوحت بين 200 الى 350 م، وضغط تراوح من 3 الى 15 ضغط جوي، وبسرع فراغية تراوحت من 0.5 الى 2.5 سا⁻¹، وكانت نسبة الهيدروجين الى النافثا 300 كنسبة حجمية.

اظهرت النتائج ان التحول للنافثا العراقية الخفيفة يزداد بزيادة درجة الحرارة ويقل بزيادة السرعة الفراغية. تكونت الازمرات ذات الرقم الاوكتاني بدرجة حرارة قليلة عند 240 م. تحسنت انتقائية تكوين الازمرات باستعمال الهيدروجين بزيادة ضغط التفاعل ولغاية 15 ضغط جوي. كانت فعالية العامل المساعد ثابتة على الدوام بضغط 15 ضغط جوي ولمدة 20 ساعة.