

Hydroisomerization of n-Pentane over Pt/Mordenite Catalyst: Effect of Feed Composition and Process Conditions

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Abstract

The hydroisomerization of pure n-pentane over H-mordenite supported Pt-catalyst was investigated in a fixed bed reactor by changing reaction parameters such as temperature, pressure, and WHSV, as well as the H₂/HC ratio. The maximum yield of isopentane over Pt/mordenite catalyst was achieved at 220 °C and a relatively low reaction pressure. To address the effect of feed composition on the catalytic performance of the samples, the catalysts were assessed for activity and selectivity in the isomerization of a mixture consisting of n-pentane (70 wt.%) and isopentane (30 wt.%) at 220 °C. The effects of pressure, WHSV, and H₂/HC ratio on the catalyst performance were also studied using binary mixtures of the pentane isomers as a feedstock. It was observed that an effect of WHSV and H₂/HC on the catalytic performance was similar to its behavior in pure n-pentane isomerization, while the conversion of n-pentane in the binary mixture showed a different trend and had a minimum value at 1.5 bar. It could be due to the presence of isopentane in feed and adsorption phenomenon of binary mixture on mordenite-supported catalyst.

Keywords: Pentane Isomerization, Pt/Mordenite, Process Conditions, Feed Composition

1. Introduction

Due to the increasing demand of isoalkanes the catalytic isomerization of linear alkanes to branched ones is an industrially important reaction, considered as an efficient alternative for octane boosters instead of oxygenates and aromatic compounds which are subjected to strict environmental restriction (Al-Kandari et al., 2009; Kamarudin et al., 2012; Ramos et al., 2005; Villegas et al., 2006). Gasoline containing high quantity of linear-chain compounds has a low octane number. However, such a content can be increased when gasoline is subjected to an isomerization process through which linear-chain molecules are converted into ramified molecules (Talebi et al., 2008; Viswanadham et al.; Yoshioka et al., 2005). The isomerization processes involve acid or bifunctional metal acid catalysts (Caeiro et al., 2006; Setiabudi et al., 2012b). Chlorinated alumina and zeolites have been developed for the isomerization of C₅-C₆ (Essayem et al., 2003). Additional research has been carried out on Pt/sulfated

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ZrO₂ and Pt/WO_x-ZrO₂ catalysts (Chu et al., 1998). Zeolites are strong solid acids, which are able to initiate the alkane conversion at a relatively low temperature thermodynamically favoring the branched isomer formation (Valyon et al., 2002). To achieve maximum isomer yields, the isomerization of C₅–C₆ paraffins must be carried out at the lowest possible temperatures over highly efficient catalysts (Essayem et al., 2003).

These catalysts are comprised of a metal component for the dehydrogenation/hydrogenation, which is provided by a noble metal basically platinum or palladium, and an acid function generated by zeolites like mordenite, Beta, and ZSM-5 for isomerization/cracking (Kumar et al., 2007; Soualah et al., 2010). According to the classical isomerization mechanism, paraffins are dehydrogenated on the catalyst metal sites, and the produced olefins are protonated on the Bronsted acid sites to the corresponding alkylcarbenium ions. These carbenium ions undergo skeletal rearrangement and β-scission followed by deprotonation and hydrogenation over metal to the corresponding paraffins (Deldari, 2005).

The isomerization of pentane and hexane is successfully carried out using Pt on zeolites catalysts (Bogdan et al., 2007; Chen et al., 2006; Jiménez et al., 2003; Kusakari et al., 2002; Lenoir et al., 2005; López et al., 2010; Miyaji et al., 2002; van de Runstraat et al., 1997; Yashima et al., 1996; Zhang et al., 1995). However, difficulties are encountered with a mixture of linear and branch hydrocarbons; because of thermodynamic equilibrium restrictions, the linear molecules cannot fully be converted to the desired branched molecules. On the other hand, the isomerization of the n-pentane and n-hexane in virgin naphtha appears to be the major gasoline octane improvement process. Light virgin naphtha consists of linear and branch C₅-C₆, so a study on the isomerization of C₅-C₆ isomers mixture is necessary. Hollo et al. (Holló et al., 2002) studied the kinetic isomerization of n-pentane and n-hexane mixture using a Pt-HMOR catalyst. Jiménez et al. (Jiménez et al., 2003) examined the hydroisomerization of a mixture of n-hexane and n-heptane on various catalysts consisting of platinum supported on different types of zeolite. However, to the best of our knowledge, there are no data available in the literature correlating to isomerization of pentane or hexane isomer mixtures. Therefore, a study on this subject is necessary.

The purposes of the present study are (i) further exploring the behavior of Pt/mordenite zeolites as catalysts for the isomerization of pure n-pentane and (ii) using the results from the first part and relating the catalyst systems to the isomerization of pentane isomers mixture. To the best our knowledge, this topic has not previously been described.

2. Experimental

2.1. Preparation of the catalysts

Pt/mordenite zeolite catalyst was prepared using a commercially available mordenite zeolite from Zeolyst. The nominal platinum concentration was 0.5 wt.%. In the impregnation method, 1.00 g of the mordenite zeolite was impregnated with the minimum amount of H₂PtCl₆ solution required to wet the solid and stirred for 24 hrs at ambient temperature. Then, the solvent was removed by evaporation at 100 °C for 12 hrs. Finally, the samples were carefully calcined at a heating rate of 0.5 °C/min to 623 K in a flowing air and maintaining this temperature for 3 hrs.

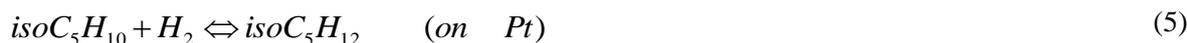
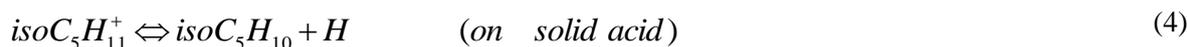
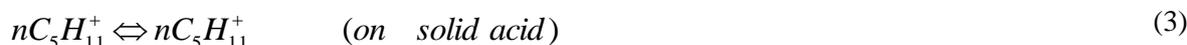
2.2 Characterization

The XRD patterns of MOR zeolite were determined using a TW3710 Philips X'Pert diffractometer using CuKα radiation as a filter (λ=1.54 Å). The data were collected within the 2θ range of 5° and 50° at a 0.02° 2θ-step and 2 s per step (40 kV and 30 mA). X-ray diffraction (XRD) was performed to

identify the product crystals phase. The morphology and size of zeolite catalysts were investigated using scanning electron microscopy (SEM, LEO 440I, 3×105, LEO, UK). The BET (Brunauer Emmette Teller) surface area of the catalysts was determined by N₂ physisorption at 77 K using a Quanta chrome chembet-3000.

2.3. Reactions, reactor system, and product analysis

The reaction network for the alkane isomerization involves many parallel and consecutive hydrogenation, isomerization, alkylation, and cracking. Main reaction pathway is as follows (Ono, 2003):



The alkane is dehydrogenated on metallic sites to the corresponding alkene, which is isomerized by acid sites into a branched alkene. The branched alkene is then hydrogenated into the branched alkane again on the metallic sites.

Figure 1 shows the schematic drawing of the experimental setup. A stainless-steel flow-type tubular reactor (internal diameter = 5 mm and length = 4.5 cm) which contains 0.5 g of a catalyst diluted with inert nonporous silica-glass, possessing the same dimension of the catalyst particles, was used in all the hydroconversion runs. The reactor was heated in an electrical furnace. Hydrogen gas was used as a carrier and simultaneously as a reactant in the reactions under study. The organic feed was pumped into the system by a syringe pump (Fanavaran Nano-Meghyas, model SP. 1000) that allowed slow, constant flow rates. The gaseous reaction effluent was analyzed using on-line gas chromatograph with a flame ionization detector (Teif Gostar Co.) and a capillary column (Cat. No. TR-110222, Serial No.: p2085307, TRB-1, Tecknokroma, 1:25 m, ID:0.25).

The experimental data was collected over a wide range of experimental conditions, including pure and binary mixture of pentane isomers as a feed stock, temperature range of 150–350 °C, pressure range of 0–2 bar, H₂ to hydrocarbon (HC) molar ratio of 7–50, and WHSV range of 0.1–0.8 hr⁻¹.

The total conversion of the n-pentane ($X_{n\text{-pentane}}$) was calculated according to Equation 6:

$$X_{n\text{-pentane}} = \frac{\sum A_i - A_{n\text{-pentane}}}{\sum A_i} \times 100 \quad (6)$$

And for the reaction product, or the asset of the products, the selectivity (S) is defined by Equation 7:

$$S_i = \frac{A_i}{\sum A_i - A_{n\text{-pentane}}} \times 100 \quad (7)$$

where, A_i is the corrected chromatographic area for a particular compound used to express the conversion and selectivity as molar percentages (López et al., 2008).

Yield (Y_i) to a particular product was calculated according to Equation 8 (Setiabudi et al., 2012a).

$$Y_i = \frac{X_{n\text{-pentane}} \times S_i}{100} \quad (8)$$

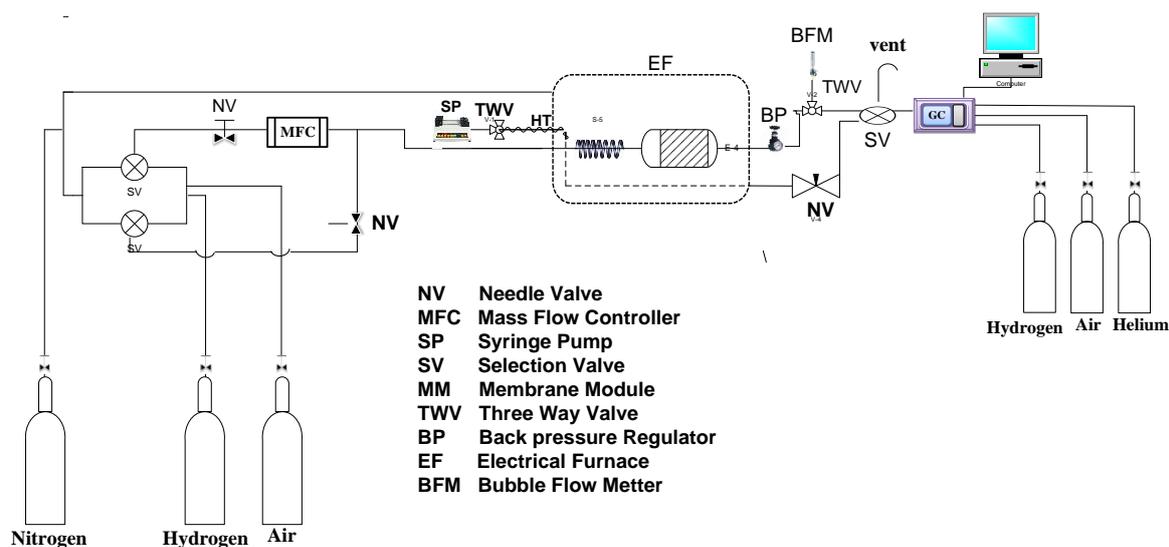


Figure 1

A schematic illustration of the experimental setup for the reaction experiments.

3. Results and discussion

Figure 2 shows the SEM micrograph of the Pt/mordenite zeolite catalyst. The image indicates that the catalyst has a homogeneous morphology. The surface area is a key factor in the catalyst activity. A high surface area improves the adsorption of reactant. The specific surface area of the catalysts was measured by the BET method. The surface area of Pt/mordenite zeolite was 296.69 m²/g. The XRD pattern of Pt/mordenite zeolite (Figure 3) exhibits the most intense diffraction peaks at $2\theta = 6\text{--}30^\circ$, and it thus confirmed the MOR structure of zeolite as well as its good crystalline nature.

The hydroisomerization of pure n-pentane and n-pentane in a binary mixture of pentane isomers was performed over the Pt/mordenite catalyst in a wide range of experimental conditions. The hydroconversion products consist of both isomerization and cracking products. Hence, in the following subsections, the effects of reaction parameters on the catalytic performance of pure n-pentane as the feed are demonstrated by catalytic activity and isomerization selectivity. Then, the isomerization of n-pentane in the binary mixture is discussed in the last part of this section.

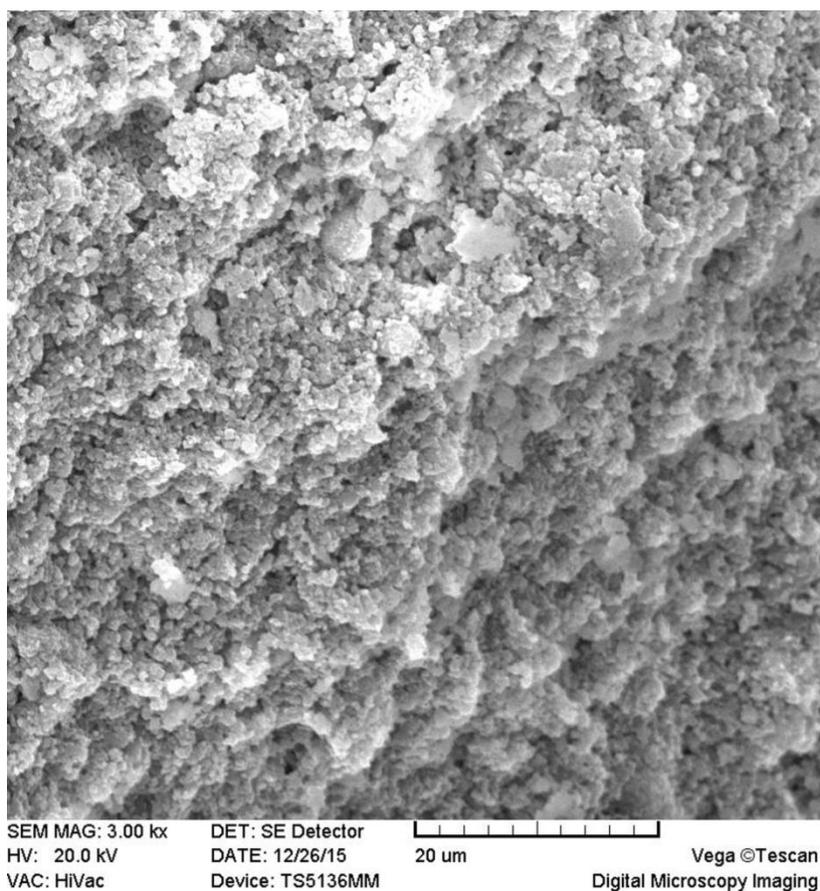


Figure 2:
SEM picture of Pt/MOR zeolite catalyst.

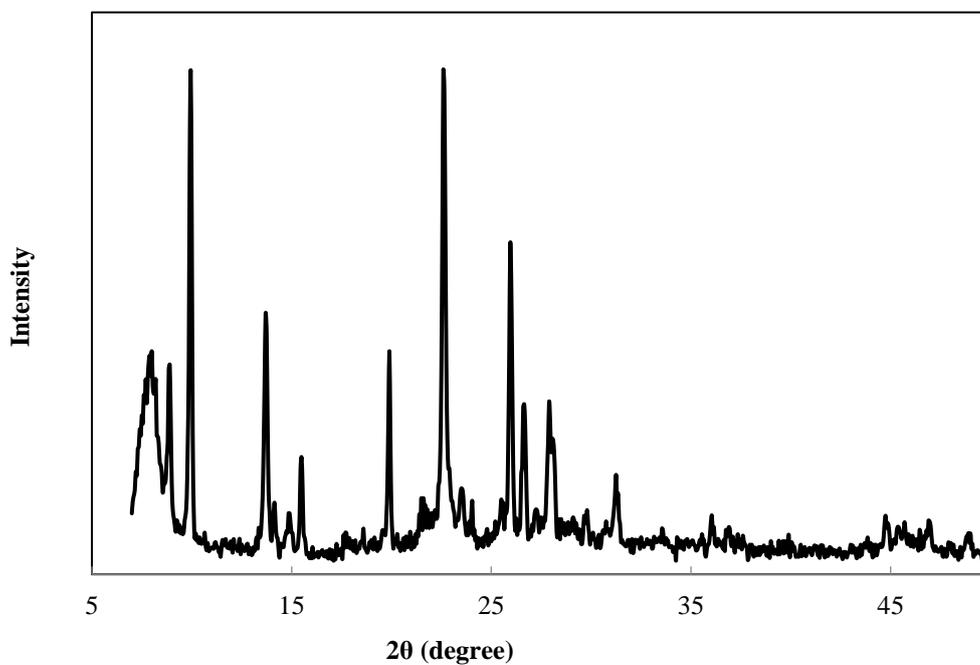


Figure 3
XRD diffractogram of Pt/MOR zeolite catalyst.

3.1. Isomerization of pure n-pentane

The isomerization experiments were carried out starting with the pure n-pentane as the feed stock using the Pt/mordenite catalyst.

3.1.1. Effect of reaction temperature

Figure 4 shows the conversion of n-pentane as a function of reaction temperature. The tests were performed in H_2 at temperatures ranging from 150 to 350 °C and atmospheric pressure. It can be clearly seen that the catalyst showed a high catalytic activity for the isomerization of n-pentane, particularly in the temperature range of 220-350 °C. Because of the low activity of the catalyst and the low reactivity of n-pentane, the conversion of n-pentane is negligible for temperatures below 180 °C. By increasing the temperature from 180 to 220 °C, the conversion of n-pentane increased greatly; however, a further increase in temperature results in a slow rise in conversion. This can be attributed to an increase in the number of sites which can be activated for the reaction when the temperature increases in the range of 180-220 °C; however, the rate of increase in conversion declines because of thermodynamic restriction at higher temperature. In other words, an increase in temperature always corresponds to an increase in the reaction rate. Thus, at low temperatures, the actual conversion will be far below the equilibrium conversion because of low reaction velocity. On the contrary, at higher temperatures, the equilibrium conversion will be more easily reached due to a high reaction rate. Consequently as mentioned in the literature (Yasakova et al., 2010), the yield of isoparaffins is limited by the thermodynamic equilibrium at higher temperatures, while, at lower temperatures, it is limited by the low reaction rate (kinetic limitation).

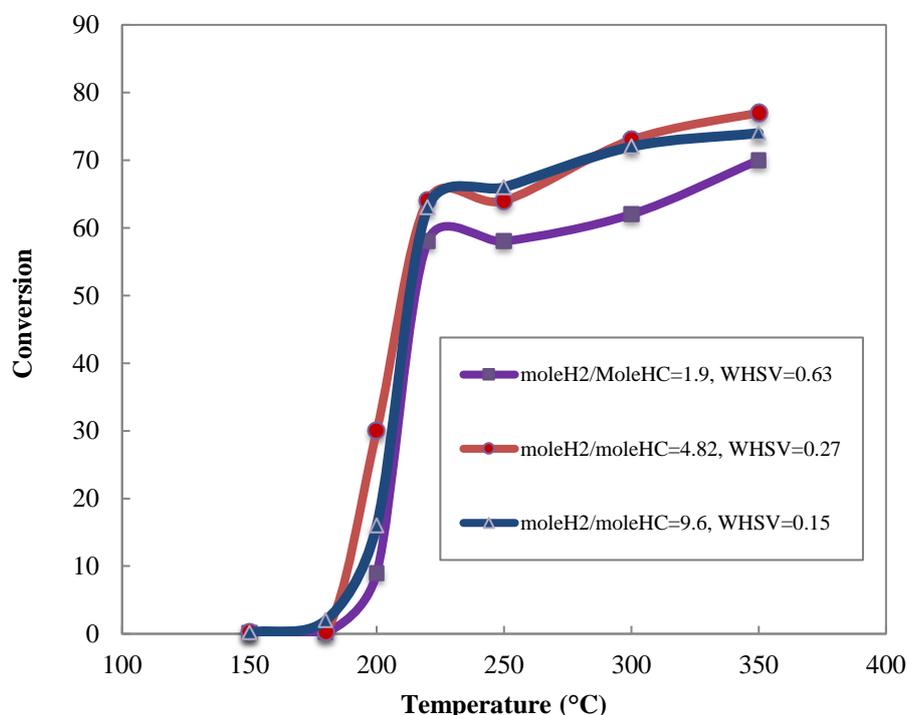


Figure 4

Conversion of n-C₅ as a function of temperature over Pt/mordenite at atmospheric pressure.

The isomerization selectivity on Pt/mordenite zeolite catalyst was illustrated in Figure 5. Isopentane selectivity was high and the selectivity was decreased with an increase in reaction temperature. The

cracking reaction was preferred in a high temperature region. Therefore, the selectivity was decreased dramatically because of the large increase in cracking products.

Cracking at high temperatures over zeolite could be explained on the acidity and pore size within the structure. In this way, strong acidity and a narrower pore diameter will make the average life time of the carbocations on the surface longer, and thus the diffusion of the branched product becomes slower. Both factors will favor the cracking of the tertiary carbocations formed during the isomerization before they can be desorbed, and the readsorption and cracking of the branched paraffins before they leave the pores and come into the gas stream (Chica et al., 2001).

Moreover, the influences of H_2/n -pentane molar ratio on the catalytic performance of zeolite catalyst in the hydroisomerization of n -pentane are shown in Figures 4 and 5. With an increase in H_2/n -pentane, the selectivity toward isomerization increases, while the conversion is observed to decrease gradually. These results imply that H_2 affects the selective formation of isopentane to some extent.

The influence of WHSV on the catalytic behavior of Pt/mordenite zeolite is also presented in Figures 4 and 5. The conversion of n -pentane decreased slowly with an increase in WHSV, whereas the cracking products increased. At low values of WHSV, the increase in the cracking by raising WHSV was low, while it rose at high values of WHSV considerably.

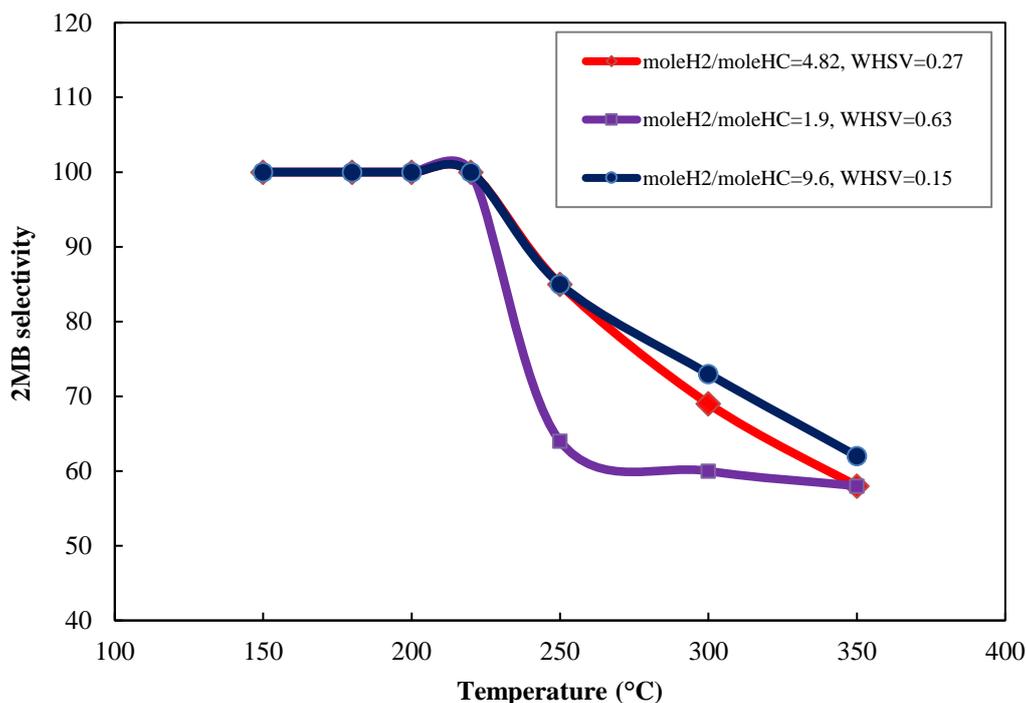


Figure 5

Selectivity of isopentane as a function of temperature over Pt/mordenite at atmospheric pressure.

The isomerization yield for n -pentane with Pt/mordenite zeolite catalyst is displayed in Figure 6. It can be seen that the isomerization yield goes through a maximum at 220 °C while the reaction temperature increases. This maximum in the isomerization yield is due to the coupling of the conversion of n -pentane and the isopentane selectivity.

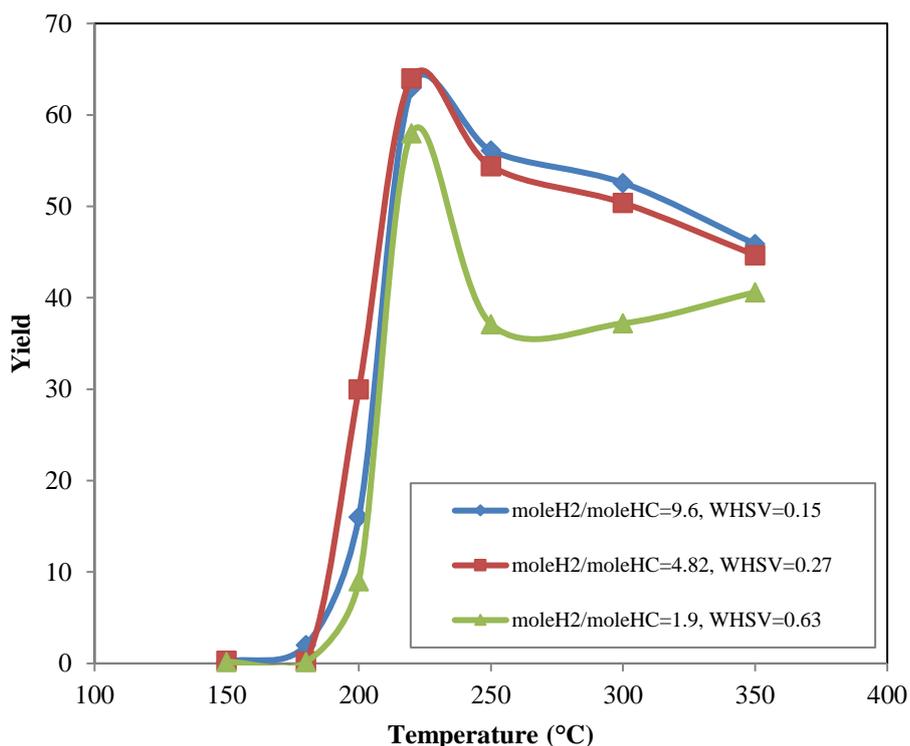


Figure 6

Effect of temperature on n-pentane isomerization yield over Pt/mordenite at atmospheric pressure.

The influences of H₂/n-pentane molar ratio on the catalytic performance of zeolite catalyst in the hydroisomerization of n-pentane are shown in Figures 4 and 5. With increasing H₂/n-pentane, the selectivity for isomerization increased, while the conversion was observed to decrease gradually. These results imply that H₂ has a significant effect on the selective formation of isopentane, which is in accordance with the kinetic models presented in the literature.

3.1.2. Effect of reaction pressure

Figure 7 shows that n-pentane conversion depends on the reaction pressure. At a reaction temperature of 250 °C, n-pentane conversion and the cracking products show a similar trend, and they are increased with increasing reaction pressure. Therefore, due to the availability of cracking products at higher reaction pressures, the selectivity toward iso-pentane was slightly lower than the one at lower setting pressures (see Figure 8). However, the pressure can suppress the side hydrogenolysis reaction at pressures higher than 10 bar.

On the other hand, n-pentane conversion slightly decreased as the reaction pressure was increased at a reaction temperature of 220 °C. The reaction pressure had no significant effect on iso-pentane selectivity. The reason for such a behavior can be due to negligible cracking reactions at low reaction temperatures (220 °C) compared to high reaction temperatures, i.e. 250 °C.

From the above results, it can be concluded that that the optimum temperature for pentane isomerization using this catalyst is 220 °C, resulting in an acceptable conversion and very high selectivity.

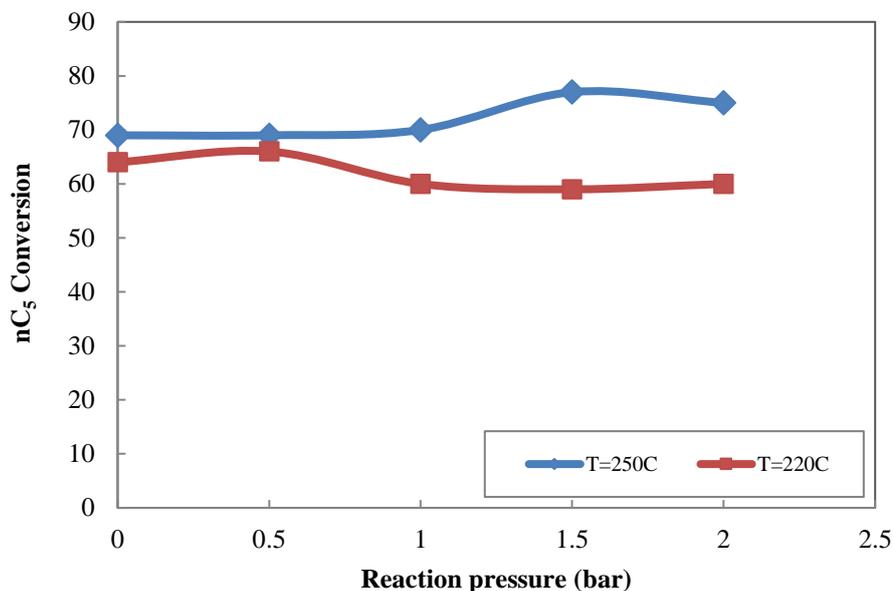


Figure 7

Conversion of n-C₅ as a function of pressure over Pt/mordenite at WHSV=0.27 hr⁻¹ and molar ratio of H₂:HC=4.82.

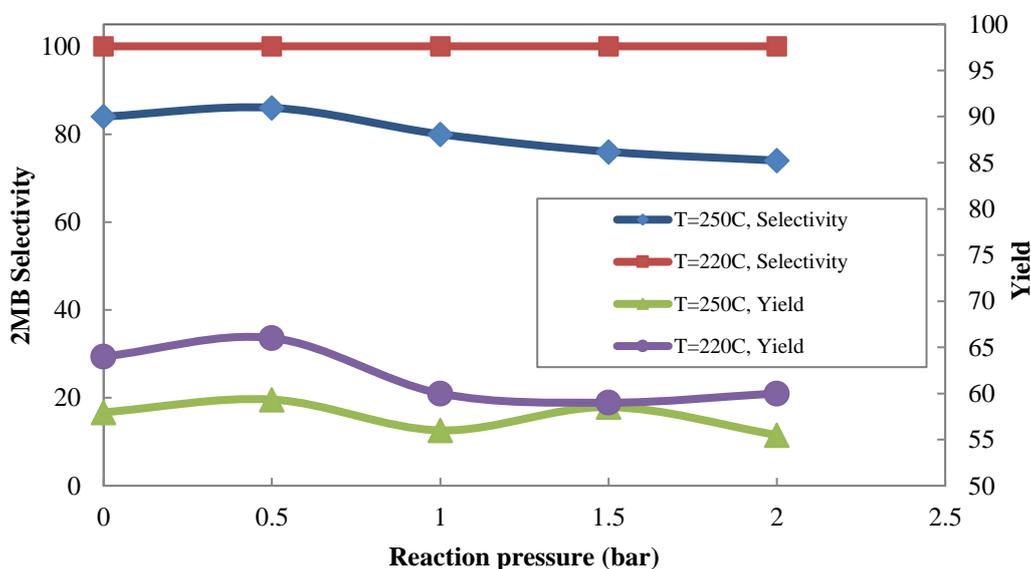


Figure 8

Selectivity of isopentane and isomerization yield as a function of pressure over Pt/mordenite at WHSV=0.27 hr⁻¹ and molar ratio of H₂:HC=4.82.

3.2. Isomerization of n-pentane in pentane isomer mixture

3.2.1. Effect of reaction pressure

To investigate the effect of isopentane in the feed stock on the isomerization process, a binary mixture

(nC₅ 70% and isopentane 30%) was fed to the isomerization reactor packed with Pt/mordenite zeolite catalyst. The representative results of n-pentane in binary mixture isomerization over Pt/mordenite are given as follows.

In Figure 9, the conversion of n-pentane in binary pentane isomers at WHSV=0.3 is plotted as a function of total reaction pressure. As can be seen, n-pentane conversion decreased with increasing the reaction pressure for all molar ratios. Normal pentane conversion reached a minimum value at 1.5 bar, and it then increased with increasing the reaction pressure. This behavior was different from the trend observed in the pure n-pentane isomerization. It can be due to the presence of isopentane in the feed and the adsorption phenomenon of the binary mixture on the mordenite catalyst. Because of the limitations of the existing syringe pumps, pressure could not be increased above 2 bar. It is likely that conversion is much higher at pressures above 2 bar.

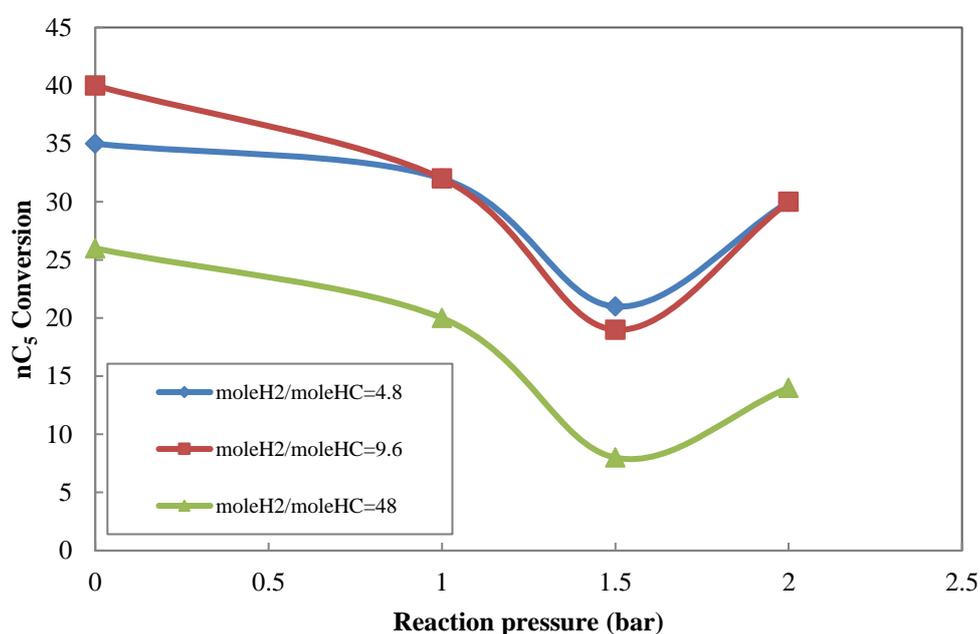


Figure 9

Conversion of n-C₅ in a binary mixture as a function of pressure over Pt/mordenite at 220 °C and WHSV=0.3 hr⁻¹.

The selectivity toward isopentane as a function of reaction pressure at a temperature of 220 °C for three molar ratios of H₂/HC is shown in Figure 10. It is apparent from Figure 8 that the reaction pressure had a very little effect on the selectivity to isopentane. At all H₂/HC molar ratios, the selectivity to isopentane varied only 1 or 2 units regardless of the reaction pressure. It can be due to reaction temperature since cracking reactions do not occur at this temperature. This result is consistent with the results of pure n-pentane isomerization at 220 °C. Moreover, the variation of the isopentane yield with reaction pressure for the Pt/mordenite catalyst sample is shown in Figure 11. As can be seen, isopentane yield showed the same trend in conversion with respect to reaction pressure over the Pt/mordenite catalyst at a temperature of 220 °C, which is, in accordance with the isopentane selectivity (100%) at 220 °C and Equation 3..

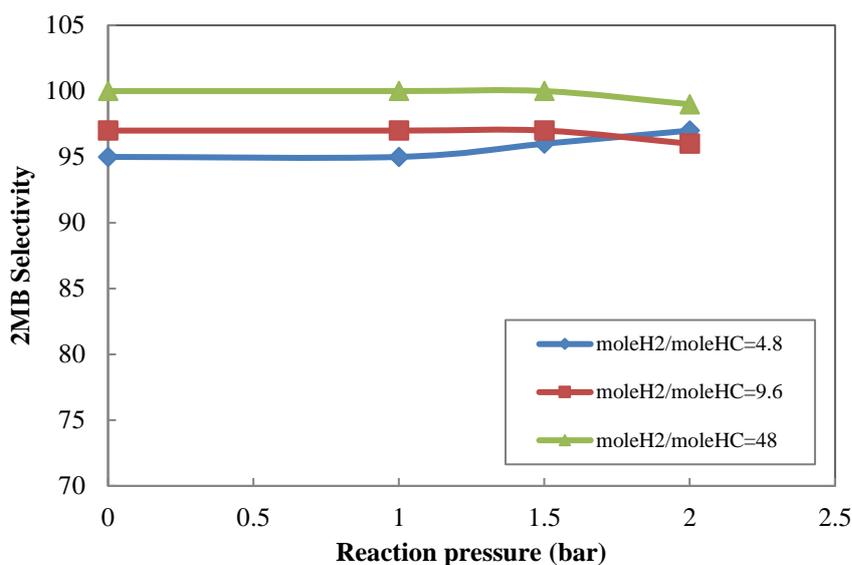


Figure 10

Selectivity of isopentane in the hydroisomerization of n-C₅ in a binary mixture as a function of pressure over Pt/mordenite at 220 °C and WHSV=0.3 hr⁻¹.

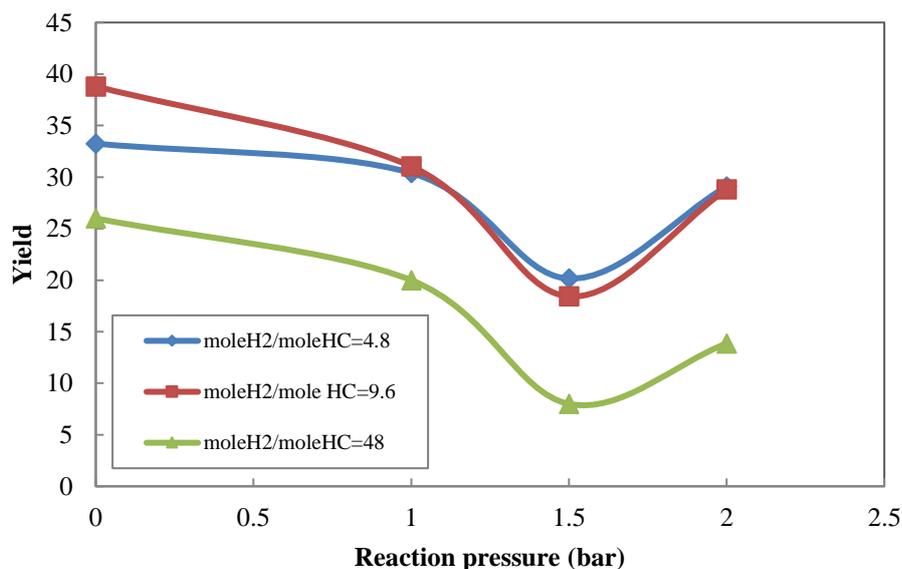


Figure 11

Yield of hydroisomerization of n-C₅ in a binary mixture as a function of pressure over Pt/mordenite at 220 °C and WHSV=0.3 hr⁻¹.

3.2.2. Effect of contact time

Along with temperature, contact time was the most important process parameter. Contact time was calculated as the residence time in the reaction space of a unit volume of reactants in the reaction conditions. At a reaction temperature of 220 °C, a reaction pressure of 1-2 bar, and a constant H₂/HC molar ratio, the effect of WHSV on n-C₅ hydroconversion in a binary mixture of pentane isomers was investigated by proportionally changing the flow rate of H₂ and HC mixture. Figures 12-14 present the influence of WHSV on the catalytic behavior of Pt/mordenite zeolite. As can be seen, the conversion

of n-C₅ and the yield of feed isomers decreased with increasing WHSV, while no significant isomerization selectivity changes were observed. With an increase in WHSV, the contact time of feed on the catalyst decreased, and thus the decrease in conversion was expectable.

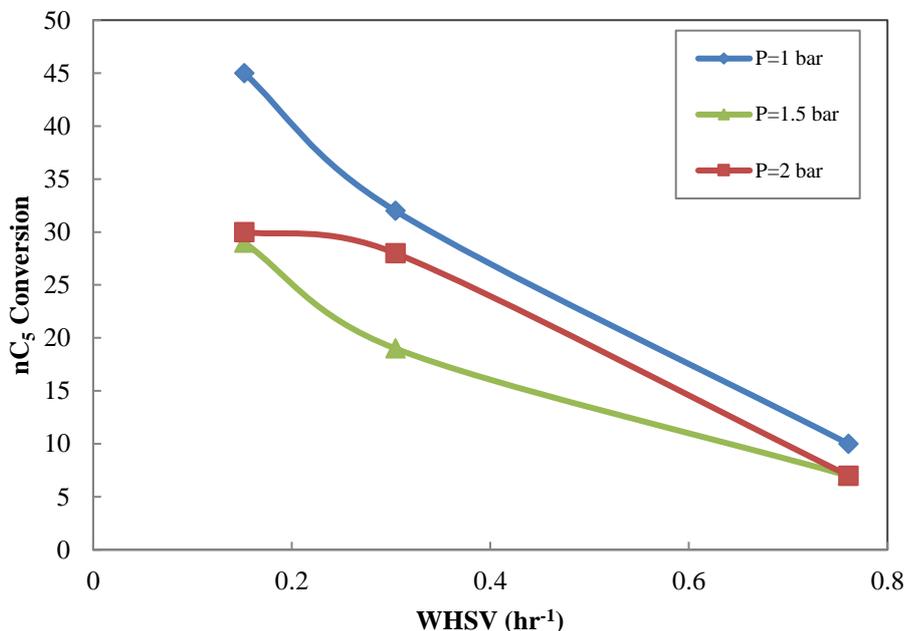


Figure 12
Effect of WHSV on n-C₅ conversion in a binary mixture over Pt/mordenite at 220 °C and molar ratio of H₂:HC=9.6.

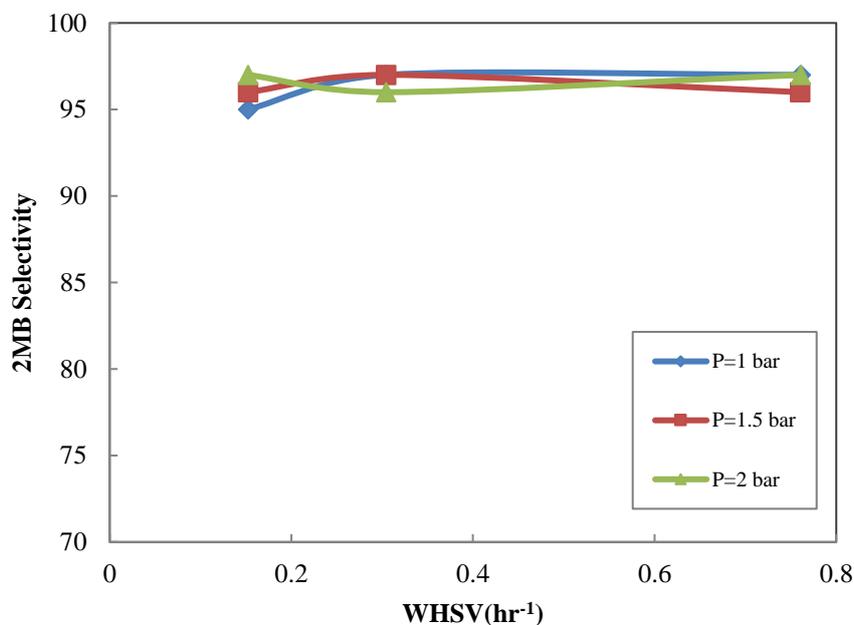


Figure 13
Effect of WHSV on selectivity of isopentane in hydroisomerization of n-C₅ in a binary mixture over Pt/mordenite at 220 °C and molar ratio of H₂:HC=9.6.

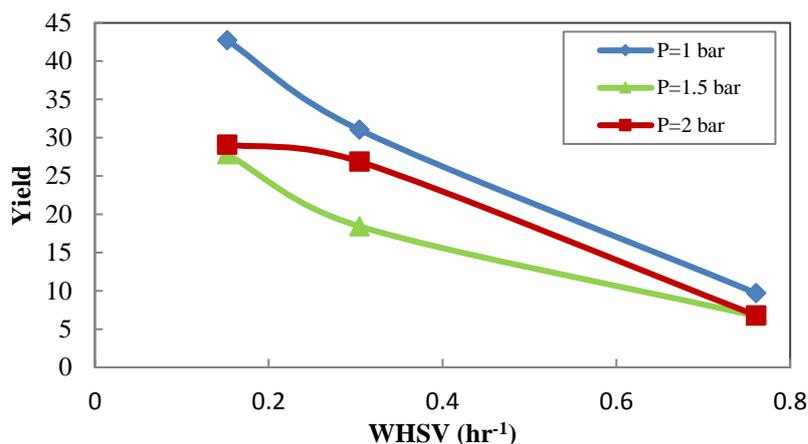


Figure 14

Effect of WHSV on yield of hydroisomerization of n-C₅ in a binary mixture over Pt/mordenite at 220 °C and molar ratio of H₂:HC=9.6.

3.2.3. Effect of H₂:HC molar ratio

The influence of H₂/HC molar ratio on the hydroconversion of n-pentane in a binary mixture of pentane isomers was investigated at a constant reaction temperature of 220 °C, reaction pressures of 0.5-1 bar, and WHSV values of 0.2 and 0.3 hr⁻¹ by changing the flow rate of H₂. As shown in Figure 15, the conversion of n-C₅ decreased with increasing the partial pressure of hydrogen (the H₂/HC mole ratio from 7 to 50), while the isomerization selectivity increased. These reflected the importance of H₂ rule in the isomerization process. The function of H₂ could be interpreted as facilitating isomerization by suppressing cracking reactions and keeping the catalytic activity high by impeding coke formation.

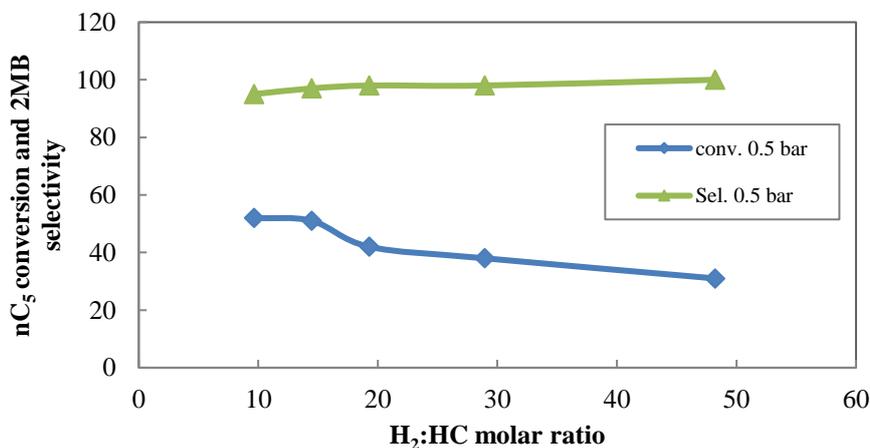


Figure 15

Effect of H₂:HC molar ratio on n-C₅ conversion and selectivity of isopentane in hydroisomerization of n-C₅ in a binary mixture over Pt/mordenite at 220 °C; (a) P=0.5 bar and WHSV=0.2 hr⁻¹ and (b) P=1 bar and WHSV=0.3 hr⁻¹.

A comparison between our results and those reported by Chica et al. (2001) is shown in Figure 16. The trends in conversion and selectivity are similar, but there is a little difference in values, which is due to the differences in pressure, WHSV, and molar ratio of H₂:HC.

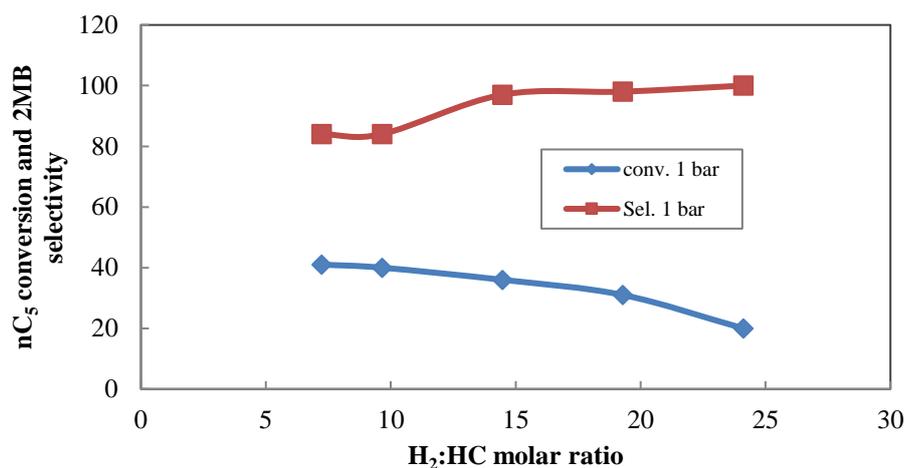


Figure 16

Comparing between results of this work (WHSV=0.15 hr⁻¹, molar ratio of H₂:HC=9.6, and atmospheric pressure) and Chica et al. (WHSV=5.13, molar ratio of H₂:HC=15, and pressure=20 bar).

4. Conclusions

The Pt/mordenite showed a high catalytic activity in the isomerization of n-pentane, particularly in the temperature range of 220–350 °C. The cracking reactions were preferred in a high temperature region (>250 °C). With the increase of H₂:HC molar ratio, the selectivity toward isomerization increased, while the conversion was observed to decrease gradually. The conversion of n-pentane decreased slowly with an increase in WHSV, whereas the cracking products increased. The maximum yield of isopentane was achieved with the Pt/mordenite catalyst at 220 °C and at relatively low reaction pressures. The catalysts were assessed for activity and selectivity in the isomerization of n-pentane in a mixture consisting of n-pentane (70 wt.%) and isopentane (30 wt.%) at 220 °C. Conversion of n-pentane changed with the reaction pressure and reached a minimum value at 1.5 bar; this behavior is different from the trend observed in the pure n-pentane isomerization, which can be due to the presence of isopentane in feed and the adsorption phenomenon of the binary mixture on Pt/ mordenite catalyst. The conversion of n-C₅ and the yield of feed isomers decreased with increasing WHSV, while no significant isomerization selectivity changes were observed. With respect to these results, the investigation of pentane isomerization at a high pressure and the study of the kinetic of pentane isomerization are suggested for future works.

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Nomenclature

| | |
|---|----------------------------------|
| X | : n-pentane conversion |
| A | : Corrected chromatographic area |
| Y | : Yield of reaction |
| S | : Selectivity of reaction |

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