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HYDROISOMERIZATION OF n-HEXANE USING UNLOADED AND LOW PLATINUM LOADED H-ZSM-5 AND H-MOR CATALYSTS

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ABSTRACT

The applicability for investing and comparing two zeolites (H-ZSM-5 and H-MOR) as supports for low-platinum, less-expensive, catalysts for the hydroisomerization of n-paraffins in petroleum naphtha is investigated. n-Hexane was used as a model n-paraffin feed at temperatures of 250-500°C in a pulsed reactor in hydrogen flow at atmospheric pressure. The H-ZSM-5 zeolite acquires higher density of strong acid sites which are still less strongly acidic than H-MOR. These somewhat milder acid sites enhance hydroisomerization after incorporating 0.15%Pt. Moreover, hydrochlorination and hydrofluorination of the 0.15%Pt/H-ZSM-5 catalyst were carried out for modifying its acidity hoping to maximize its hydroisomerization activity. Furthermore, the effect of this hydrohalogenation treatment on other hydroconversion reactions (hydrocracking and dehydrocyclization) was investigated. The HCl treatment has significantly enhanced the required catalytic functions, whereas HF appears mostly deteriorative.

KEY WORD: n-hexane, hydroisomerization, hydrocracking, dehydrocyclization, H-ZSM-5, H-MOR, hydrohalogenation.

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1. INTRODUCTION

The hydroisomerization of n-paraffins is a process of prime importance for the upgrading of paraffinic low-octane naphtha for producing high-octane C_5^+ gasoline. Various modifications have been carried out to improve the hydroisomerization catalysts in this laboratory. A great effort has been directed to modify zeolite supports in these catalysts via strengthening or creating stronger acid sites, compared to the traditionally used γ-alumina catalytic support.

The hydroisomerization of n-hexane has been studied using 0.35%Pt supported on H-MOR or NH4-MOR steamed before or after Pt incorporation [1,2]; where maximum hexane isomers amounted to 59.0% at 275°C. Shifting to using H-ZSM-5 zeolite support loaded with 0.30%Pt gives 71.8% isohexanes at 325°C when dealuminated with EDTA [3]. Again, n-hexane was isomerized on Pt/H-MOR and Pt/BEA catalysts with various Pt contents and bimetallic Pt-Pd/H-MOR, Pt-Pd/H-BEA, Pt-Ir/H-MOR and Pt-Ir/H-BEA, with various secondary metal contents [4]. The addition of Pd (activation promoter) to Pt has increased the metals dispersion in both zeolites up to a certain content, whereas Ir (promoter for counter-deactivation by coke deposition) has decreased the metal dispersion. n-Hexane hydroisomerization was significantly promoted with the doping of fluoride ion on catalysts containing Pt, Ir, Pt-Re and Pt-U supported on γ-alumina [5].

In the present work, a catalyst containing low-Pt content (0.15%) loaded on H-ZSM-5 has been doped with 3%HCl or HF; the former acid has been a significantly successful promoter, whereas the latter has mostly acted as an inhibitor.

2. EXPERIMENTAL

2.1. Preparation of the Unloaded Zeolite Catalysts (H-ZSM-5 and H-MOR)

The Na-form of ZSM-5 and MOR zeolites were kindly provided by Süd-Chemie AG, Germany. They were exchanged in 0.7 M NH_4NO_3 solution five times; each time with a fresh solution for 8 h at 70°C. The NH_4-zeolite obtained was separated, washed with distilled water till free of the NO_3^+ ion, dried at 110°C overnight then calcined in air at 530°C for 3h to be deammoniated to H-ZSM-5 and H-MOR. The Na^+ content was less than 0.02wt%.

2.1.2. Preparation of 0.15%Pt/H-ZSM-5 and 0.15%Pt/H-MOR catalysts

Both catalysts were prepared using the impregnation method. A quantity of chloroplatinic acid, necessary for loading 0.15wt% of Pt metal in each catalyst (0.15%Pt/H-ZSM-5 or 0.15%Pt/H-MOR) was dissolved in distilled water. This solution was adjusted to cover the zeolite in a beaker and left for 24 h at room temperature. The catalyst was then dried at 110°C overnight then calcined in air for 4 h at 530°C and reduced at 500°C in H_2 flow of 20 $cm^3\ min^{-1}$ in a flow reactor for 8h.

2.2. Hydrochlorination and Hydrofluorination of the 0.15%Pt/H-ZSM-5 Catalyst

A part of the calcined 0.15%Pt/H-ZSM-5 catalyst was doped with an aqueous solution containing 3.0wt% of HCl or HF relative to the catalysts weights. The catalysts were dried at 110°C overnight then calcined in air at 530°C for 3 h. Before carrying out the hydroconversion runs, the catalysts were heated in the catalytic reactor at 500°C for 2h in a flow of 20 cm^3min^{-1} of pure hydrogen gas. It is to be noticed that ~70% and ~84% of the Cl^- and F^- ions, respectively, (determined by ion-chromatography) of the doped haloacids, remained in 0.15%Pt/H-ZSM-5(HCl) and 0.15%Pt/H-ZSM-5(HF) catalysts, respectively, after drying, calcination and reduction. This indicates a higher stability of the fluorine-zeolite bonding than the chlorine-zeolite bonding under the applied dry conditions.

2.3. n-Hexene Hydroconversion, Reactor System and Analysis

A stainless-steel microcatalytic tubular pulse reactor, containing 0.2 g of a catalyst, was used. The reactor was thermostated to ±1°C. Hydrogen was used as a carrier gas and simultaneously as a reactant, at a flow rate of 20 cm^3min^{-1}in all runs. n-Hexane feed pulses of 1.0 µl were always injected into the carrier H_2 flow passing into the inlet of the reactor which passes to OV-101 column for analysis.

2.4. Temperature Programmed Desorption (TPD) of NH_3

The TPD procedure by Aboul-Gheit [6,7] using highly sensitive (gold sensor) differential scanning calorimetry (DSC) has been applied for detecting and evaluating the desorption of presorbed ammonia from the acid sites of the current catalysts. Ammonia was initially adsorbed on the catalyst in a silica-tube furnace till saturation then desorbed in a Mettler TA-3000 DSC unit in a continuous current of purest N_2. The thermogram obtained for each catalyst gives two peaks; a low-temperature (LT) peak corresponding to enthalpy (ΔH_{des}) of ammonia desorption from the weak acid sites of the catalyst and a high temperature (HT) peak corresponding to ΔH_{des} of ammonia desorbed from the strong acid sites. The ΔH_{des} values are proportional to the number of acid sites, whereas the peak temperature (T_{max}) correlates the acid sites strength in the catalyst (Fig. 1). The TPD data obtained to evaluate strong acid sites are given in Table 1.

2.5. Dispersion of Platinum in the Catalysts

The dispersion of Pt in the current catalysts, were determined by hydrogen chemisorption using Freel's pulse technique [8]. The calcined catalyst was heated in the furnace at 500°C for 3h at a flow of 50 cm^3 min^{-1} of ultra-pure hydrogen then using purest N_2 at 500°C (degassing). After shutting off the furnace, hydrogen was pulsed into the N_2 carrier gas till saturation, i.e, appearance of H_2 peaks equivalent to complete volumes of unchemisorbed pulses. The hydrogen uptake was calculated as hydrogen atoms adsorbed per total Pt atoms. The H/Pt stoichiometry of 1/1 has been used. The data obtained are given in Tables 1 and 2.

3. RESULTS AND DISCUSSION

3.1. Hydroconversion of N-Hexane Using Unloaded and Pt-Loaded Zeolites

The activities of the H-MOR and H-ZSM-5 zeolites as catalyst for the hydroconversion of n-hexane at temperatures of 250-500°C and under atmospheric pressure, in a pulsed type microreactor using H_2 flow of $20cm^3min^{-1}$ are shown in Fig. 2a. Evidently, H-ZSM-5 is much less active than H-MOR; the conversion of n-hexane using the former catalyst ranges between 0.8 and 35.2% at reaction temperatures of 300-500°C, respectively, whereas using the latter catalyst, conversion is 12.0- 96.1% at temperatures of 275-500°C, respectively.

The conversion of n-hexane using H-ZSM-5 catalyst is attributed only to hydrocracking reactions, where C_2, C_3 and C_4 components (iso-and n-butanes) are obtained. Iso-hexanes and benzene have not been detected among products. Methane is also assumed to be absent since C_5 components are absent. Hydrogenolysis to methane is a catalytic property of the metal function in dual-functional catalysts. However, the significantly higher activity of the H-MOR catalyst is attributed to its higher acid strength (Table 1). On this catalyst, hexane isomers are also present in trace amounts. The hydrocracked products contain iso- and n-butane as well as iso- and n-pentane, together with $C_{1+}C_2$, C_3, iso- and n-butanes. Moreover, benzene is produced at higher temperatures, i.e., 1.1-3.7% at 375-500°C, respectively.

It has to be pointed out that almost all hydrocracking products using the unloaded H-ZSM-5 and H-MOR zeolites as catalysts (in absence of metals) are saturated hydrocarbons. In the early work of Platteauw et al. [9], it has been assumed that amorphous SiO_2-Al_2O_3 catalyst possessed a “self-hydrogenating capability” accomplished via hydrogen-transfer for a period of time, even in absence of added hydrogen. Also, Aboul-Gheit et al. [10] has found that unloaded γ-alumina and H-MOR zeolite as catalysts [11] possessed self hydrogenating capability. Furthermore, Sano et al. [12] found that H-ZSM-5 zeolite is active as a hydrogenation catalyst. At 535°C, they have been able to convert $\approx$ 95% of ethene to ethane and concluded that hydrogenation takes place on the acid sites in absence of a metal. Also, Kanai et al. [13] shows that Brönsted acid sites become active hydrogenation sites of alkenes at higher temperatures. Furthermore, Chu et al. [14] state that in absence of active metal the acid centres of H-BEA zeolite themselves have hydrogenation-dehydrogenation activities.

3.1.2. Hydroconversion of n-hexane using 0.15%Pt/H-MOR and 0.15%Pt/H-ZSM-5 catalysts

The hydroconversion activity of n-hexane at temperatures of 275-500°C is found to range between 60.6 and 99.0% on a catalyst containing 0.15%Pt/H-MOR, and between 36.0 and 77.5%, respectively, on a catalyst containing 0.15%Pt/H-ZSM-5 (Fig. 2b). Hydroconversion using the H-MOR catalyst has not been significantly enhanced by platinum incorporation, compared to the activity of 0.15%Pt/H-ZSM-5 (compare Figs. 2a and 2b). n-Hexane hydroconversion includes hydroisomerization that takes place at low reaction temperatures, together with hydrocracking and dehydrocyclization to benzene at higher temperatures.

3.1.2.1 Hydroisomerization of n-hexane using 0.15%Pt/H-MOR and 0.15%Pt/H-ZSM-5 catalysts

Normally, n-paraffins are present as major components in the petroleum naphtha fractions. These paraffins acquire low octane numbers, e.g., n-hexane, n-heptane and n-octane have octane numbers of 26, zero, and a negative octane number, respectively. Hence, upgrading of straight-run naphtha via the hydroisomerization processes produces mixtures of higher-octane components and therefore, this refining process is indeed of prime commercial importance. In the present work, n-hexane isomerization occurring on the current 0.15%Pt/H-MOR and 0.15%Pt/H-ZSM-5 catalysts, produces several branched isoparaffins acquiring varying octane numbers. However, the produced isohexanes are susceptible to undergo hydrocracking which is practically considered as a loss. Therefore, these cracked products have to be minimized.

Fig. 3 shows that the hexane isomers using the 0.15%Pt/H-MOR catalyst are significantly lower than using the 0.15%Pt/H-ZSM-5 catalyst at 250°C (19.0% vs. 30.0%). On the former catalyst, the isomers increase with temperature to reach a maximum of 38.1% at 275°C, then rapidly decrease till disappear completely at 375°C. However, using the Pt/H-ZSM-5 catalyst, iso-hexanes increase gradually with temperature to reach a maximum of 41.3% at 350°C then decrease also gradually with further decreasing the temperature to reach 13.6% at 500°C. This gradual increase and decrease of isomers with temperature can be attributed to the lower acid sites strength of the H-ZSM-5 component compared to H-MOR.

3.1.2.2 Hydrocracking of n-hexane using 0.15%Pt/H-MOR and 0.15%Pt/H-ZSM-5 catalysts

Hydrocracking is a traditional competitor of hydroisomerisation. During the formation of an isomeric carbonium-ion, it may rupture (hydrocrack) before it acquires a hydride ion to form a stable isohexane molecule if the acidity strength of the catalyst and/or the temperature of reaction is high, because both isomerization and cracking reactions take place by the same mechanism (carbonium ion mechanism). Fig. 4 shows that on the 0.15%Pt/H-MOR catalyst, hydrocracking starts with 2.7% at 275°C then sharply increases with temperature to reach as high as 98.2% at 375°C, beyond which this reaction slightly slows down by a further increase of temperature to reach 95.1% at 500°C. On the other hand, using the 0.15%Pt/H-ZSM-5, hydrocracking starts very mildly (<1.0%) at 325°C and increases relatively slowly with temperature to reach 46.9% at 500°C. The hydrocracking activities of the 0.15%Pt/H-ZSM-5 and 0.15%Pt/H-MOR catalysts are compatible with the acid sites strength of these catalysts (Table 1). These data show the possible commercial applicability of the 0.15%Pt/H-MOR catalyst in petroleum fractions hydrocracking processes at relatively low temperatures.

3.1.2.3.Dehydrocyclization of n-hexane using 0.15%Pt/MOR and 0.15%Pt/H-ZSM-5 catalysts:

The dehydrocyclization of n-hexane to benzene (Fig.5) on Pt/H-ZSM-5 catalyst starts with 2.4% at 350°C and increases gradually to reach 17.1% at 500°C, whereas on the Pt/H-MOR catalyst, benzene formation is much lower. Correlation of Figs. 4 and 5 shows that although the hydrocracking activity of the Pt/H-ZSM-5 catalyst is much lower than that of Pt/H-MOR, the dehydrocyclization activity of the latter catalyst is much lower than that of the Pt/H-ZSM-5 catalyst. Although hydrocracking is primarily dependent on the catalytic acid site strength, dehydrocyclization is primarily dependent on the catalytic metallic sites. In both catalysts, the metal component is apparently the same (0.15wt%platinum). Hence, it is not the quantity of the metal that affects or control the dehydrocyclization activity. It is the state of the metallic sites in the catalyst. Primarily, the dispersion of Pt (fraction of Pt exposed) in the zeolite which amounts to 71.6% in 0.15%Pt/MOR and 79.6% in 0.15%Pt/H-ZSM-5. Nevertheless, the relatively very low Pt content in the current catalysts makes the zeolitic component much more significantly controlling.

This significantly lower activity of the Pt/H-MOR catalyst for benzene production than Pt/H-ZSM-5 catalyst can not be attributed to the effect of the window size of the zeolite, since MOR is a large pore zeolite with a ring major chemical size of twelve, whereas ZSM-5 is a medium pore zeolite with 10 membered ring. Therefore, diffusion of the aromatic ring with 6.3Å diameter cannot be the limiting step in benzene formation in neither H-ZSM-5 nor in H-MOR catalyst. However, with straight channel of the ZSM-5 zeolite, diffusion is five times faster than in its sinusoidal channel for molecules close in size to the diameter of the pores, i.e., benzene. This slower diffusion cannot be a handicap, but it will be more effective for contacting the n-hexane molecules with the largest possible number of active sites in these sinusoidal channels of ZSM-5. Furthermore, the pores of ZSM-5 are of tridirectional structure, whereas MOR acquires unidirectional channel structure [15].

The role of diffusion is aimed here to be clarified because it requires more clarification, since the Pt content in both catalysts is equal (0.15wt %), but the acid sites strength is higher in Pt/H-MOR than in Pt/H-ZSM-5 (Table 1). In the former catalyst, the strong acid sites peak covers a temperature range of 388-608°C with a maximum at 505°C, whereas in the latter catalyst this peak covers a temperature range of 332-588°C with maximum at 469°C. Basically, the higher the temperature of the ammonia desorption, the higher the acid site strength. So, the higher conversion of n-hexane to benzene on 0.15% Pt/H-ZSM-5 than 0.15% Pt/H-MOR can be attributed to the effect of difference in diffusion and adsorption of the formed benzene molecules due to hydrophilicity-hydrophobicity characteristics.

3.1.3. Effect of hydrohalogenation of the 0.15%Pt/H-ZSM-5 catalyst on its catalytic activities

Although the number of acid sites in the 0.15%Pt/H-ZSM-5 catalyst is larger than in 0.15%Pt/H-MOR, the strength of these acid sites is weaker in the former catalyst

(Table 2). Therefore, we examined the effect of hydrohalogenation of the 0.15%Pt/H-ZSM-5 catalyst on strengthening and/or increasing the number of its acid sites both analytically and catalytically on n-hexane hydroisomerization, hydrocracking and dehydrocyclization.

3.1.3.1. Effect on n-hexane hydroisomerization

Figure 6 compares the hydroisomerization of n-hexane using the 0.15%Pt/H-ZSM-5 catalyst before and after its hydrohalogenation; i.e., the untreated catalyst is compared with its hydrochlorinated and hydrofluorinated versions. Evidently, hydrochlorination is the prosperous outlet, whereas hydrofluorination results in a negative effect. The former treatment, not only enhances C_6 isomers production, but also results in lowering the temperature at which maximum isomerisation takes place (61.7% at 300°C), whereas using the untreated catalyst version, the isomers maximum is 41.0% at 350°C. Nevertheless, using the hydrofluorinated catalyst, maximum isomers dropped to 35% at a temperature of 350°C. The hydrochlorination treatment, beside being a cheap one, it also lowers the operating temperature from 350 to 300°C, which is a significantly economic addition.

The hydrochlorinating enhancement is attributed to: (1) acquiring a large number of moderately strong acid sites, strong enough for carbonium ions suitable for stable branching of the n-paraffin, (2) these acid sites, however, are not so strong that the isomerised carbonium ion doesn't crack. (3) hydrochlorination assists in increasing the dispersion of platinum (metal function in bifunctional catalysts) which is greatly important for balancing the strong acid function.

3.1.3.2. Effect on n-hexane hydrocracking

Figure 7 shows that the hydrochlorination of the 0.15%Pt/H-ZSM-5 catalyst enhances the hydrocracking of n-hexane, whereas hydrofluorination doesn't measurably affect this reaction. The larger number of acid sites, possessing a moderate strength in the 0.15%Pt/H-ZSM-5(HCl) catalyst (Table 1) gives mild cracking, particularly at the lower temperatures. Fortunately, hydrocracking of n-hexane using the current HCl treated catalyst (Fig. 6) amounts to as low as 3.5% of hydrocracked gases at 300°C. At this temperature, the maximum isomerisation occurs, which is practically an important industrial parameter. Moreover, benzene is completely absent at 300°C. Recently, the presence of benzene is medically forbidden in motor gasoline by modern environmental restrictions.

The work in this paper is primarily directed towards producing a significantly low Pt containing catalyst, cheaply treated with HCl acid. The low Pt content facilitates its high dispersion in the zeolite support, i.e., D = 86.3%, however, the untreated and the HF-treated catalysts acquire 78.6 and 65.8% dispersion, respectively.

3.1.3.3. Effect on n-hexane dehydrocyclization

The dehydrocyclization of n-hexane produces benzene at higher operation temperatures, since this reaction is an endothermic one. Using the untreated Pt/H-ZSM-5 catalyst, benzene starts production of 2.5% at 350°C and increases with temperature to reach 17.1% at 500°C, whereas using the Pt/H-ZSM-5(HCl) catalyst, benzene production starts with 1.2% at 325°C and increases as a function of temperature to reach 23.6% at 500°C (Fig. 8).

On these two catalysts, benzene formation shows some slowness at 400-500°C and 425-500°C, respectively, indicating diffusion restriction of limited extent. However, using the Pt/H-ZSM-5(HF) catalyst, benzene formation is the lowest; it increases to reach a maximum of 8.5 % at 425°C, beyond which it declines with a further increase of temperature to reach 6.3 % at 500°C. This behaviour indicates that this reaction is significantly diffusion restricted, which is attributed to the leaching of Al and Si from the zeolite framework forming fluoroalumino- and fluorosilico- amorphous debris, which is deposited in the zeolitic channels causing diffusion restrication of the reactant and products along these channels.

The enhancement of benzene production on the HCl treated catalyst is attributed to enhancement of the acid sites number and strength beside enhancing the dispersion of Pt to a significant extent. However, although HF enhances the acid site strength, the activity for benzene formation is decreased significantly due to the decrease of Pt dispersion as well as the increase of diffusion restriction by the deposited debris.

CONCLUSION

H-ZSM-5 as a catalyst for n-hexane hydroconversion is found much less active than H-MOR; the latter is highly active for hydrocracking. Incorporation of 0.15%Pt in H-ZSM-5 enhances n-hexane hydroisomerization but moderately enhances hydrocracking and dehydrocyclization. However, incorporation of 0.15%Pt in H-MOR, doesn't enhance hydrocracking, which still remaining the major reaction; dehydrocyclization is poorly enhanced, while hydroisomerization is enhanced at lower temperatures. Doping of Pt/H-ZSM-5 with HCl significantly enhances hydroisomerization, hydrocracking and dehydrocyclization, whereas doping with HF suppresses hydroisomerization and dehydrocyclization, but doesn't affect hydrocracking.

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Table 1. Ammonia TPD from the Catalysts and Pt Dispersion in the Unloaded and 0.15%Pt-Loaded Zeolite Catalysts:

Catalyst	**HT peak temperature, °C** (*)		**ΔH_{des}, Jg^{-1}** (**)	**Pt Dispersion, %**
	Range, °C	**Maximum, °C**		
H-MOR	400-620	520	72	**---**
H-ZSM-5	340-600	480	115	**---**
0.15%Pt/H-MOR	388-610	505	68	71.6
0.15%Pt/H-ZSM-5	332-588	469	95	79.6

(*) Proportional to acid sites strength (**) Proportional to acid sites number

Table 2. Ammonia TPD from the Catalysts and Pt Dispersion in the 0.15%Pt/Zeolite Catalysts before and after hydrohalogenation:

Catalyst	**HT peak temperature, °C** (*)		**ΔH_{des}, Jg^{-1}** (**)	**Pt Dispersion, %**
	Range, °C	**Maximum, °C**		
0.15%Pt/H-ZSM-5	332-588	469	95	78.6
0.15%Pt/H-ZSM-5(HCl)	337-592	474	107	86.3
0.15%Pt/H-ZSM-5(HF)	341-596	478	96	65.8

(*) Proportional to acid sites strength (**) Proportional to acid sites number

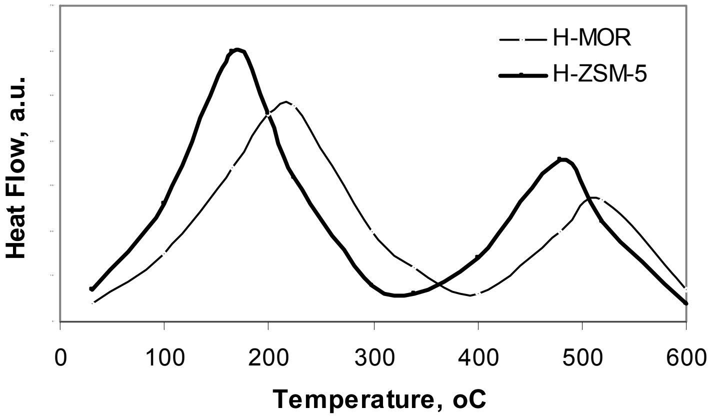

Fig. 1. TPD of ammonia from H-MOR and H-ZSM-5 zeolites

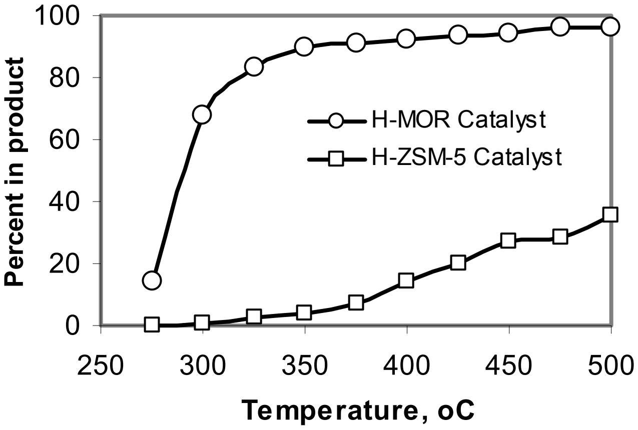

Fig. 2a. Hydroconversion of n-hexane on H-ZSM-5 and H-MOR catalysts

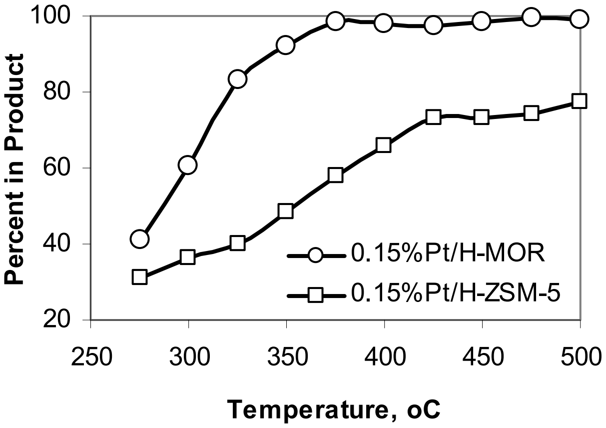

Fig. 2b. Hydroconversion of n-Hexane on 0.15%Pt-Loaded Zeolite Catalysts

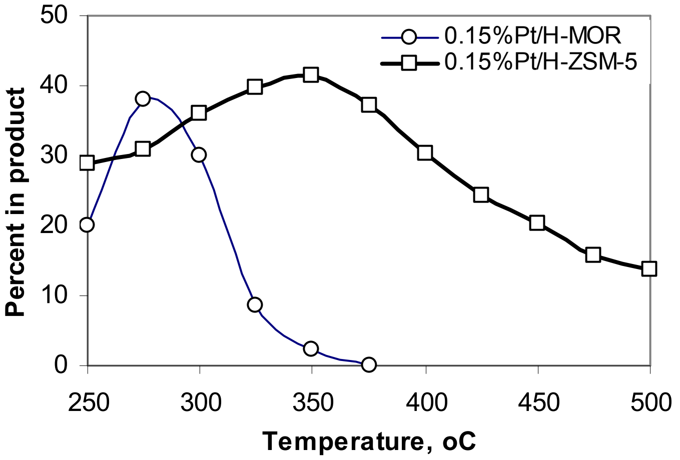

Fig. 3. Hydroisomerization of n-hexane on 0.15%Pt loaded H-MOR and H-ZSM-5 catalysts

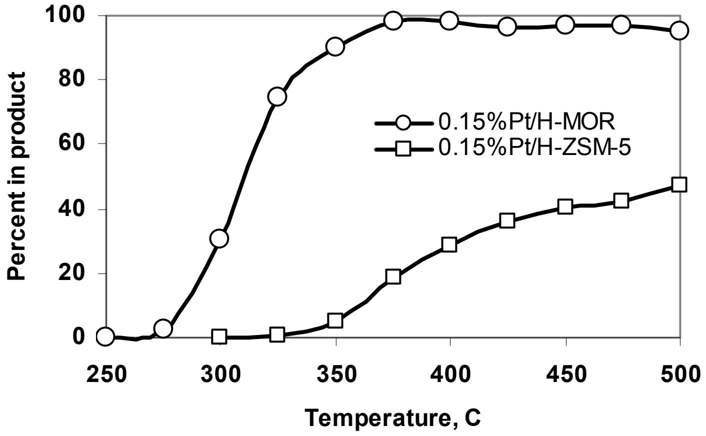

Fig. 4. Hydrocracking of n-hexane on 0.15%Pt loaded on H-MOR and H-ZSM-5 catalysts

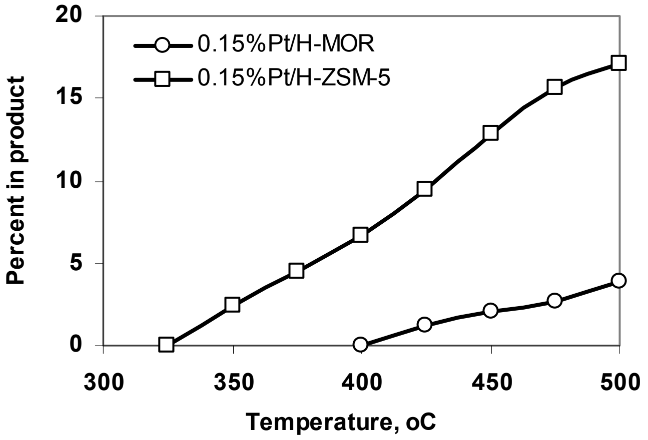

Fig. 5. Dehydrocyclization of n-hexane on 0.15%Pt loaded H-MOR and H-ZSM-5 catalysts

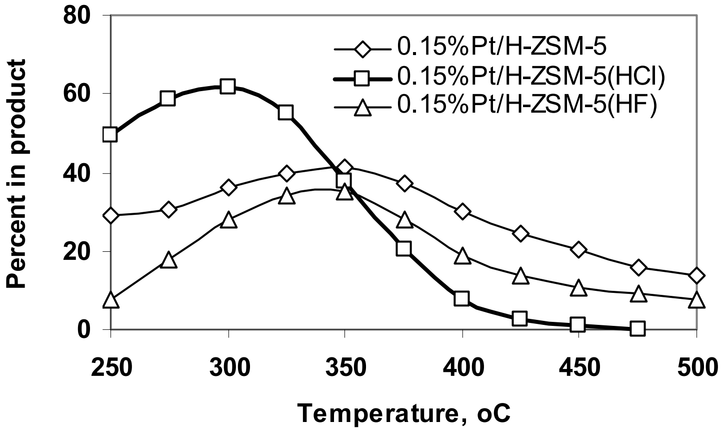

Fig. 6. n-Hexane hydroisomerization before
and after HCl and HF treatment of the catalyst

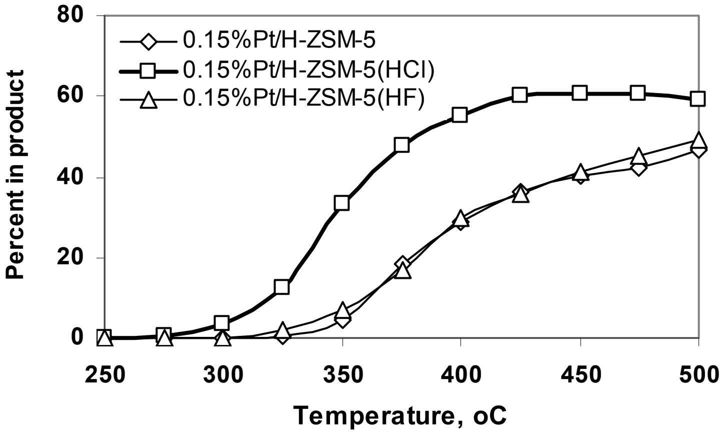

Fig.7. Hydrocracking of n-hexane before
and after HCl and HF treatment of the

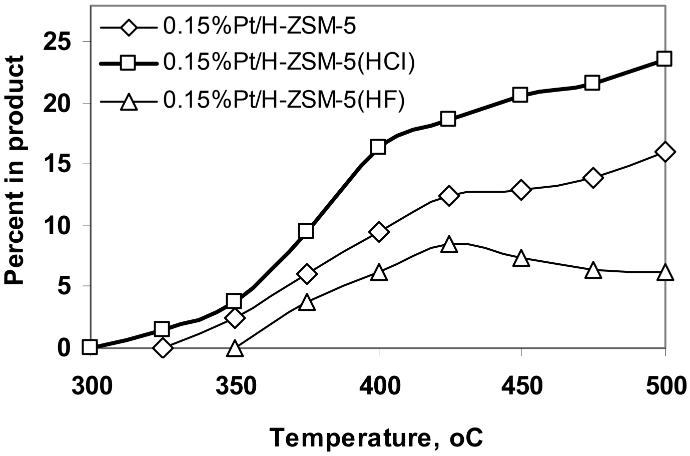

Fig. 8. Dehydrocyclization of n-hexane before
and after HCl and HF treatment of the catalyst