CHAPTER IV RESULTS AND DISCUSSION

In this work, metal loading on the mordenite was performed using two different methods (ion-exchange and impregnation methods). Metals, that were introduced into mordenite catalysts is Pt, Pd, Ga, Zn, and K. The resultant catalysts were characterized by XRD, FT-IR, XRF, BET and NH₃-TPD techniques.

4.1 Characterization of the catalysts

4.1.1 X-ray powder diffraction (XRD)

The XRD patterns of HM, and metal loaded on mordenite catalysts were shown in Figure 4.1. The mordenite in acid form (HM) shows diffraction peaks at 2 angles: 6.5, 8.6, 9.8, 13.4, 15.3, 19.3, and 22.2. For hkl planes: 110, 020, 200, 220, 310, 330, 150 and 220 [27].

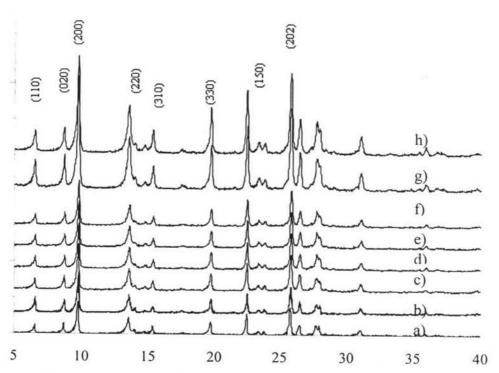


Figure 4.1 XRD patterns of mordenite and metal-loaded mordenite (a) HM, (b) 1%Pt/HM, (c) 1%Pt0.5%Ga/HM, (d) 1%Pt/1Ga%/HM, (e) 1%Pt1%Ga/K-M,

(f) 1%Pt1%Ga1%K/HM, (g) 0.5%Pt1%Zn/HM, and (h) 0.5%Pt1%Zn/HM.

For the metal-loaded mordenite samples, Pt, Pd, Ga, Zn or K, the samples exhibited X-ray diffraction patterns similar to the mordenite, which indicated that the structural of the mordenite was the same as metal/mordenite catalysts. This might be due to the small amount of metal loading. The metal containing mordenite catalysts did not show any presence of metal. It was noticed that no other phase detected.

The XRD patterns of hydrotalcite before and after calcined which prepared by alkali free coprecipitation method are shown in Figure 4.2. For the as-syn. hydrotalcite, diffraction peaks were observed at $2\theta = 11.6$, 23.4, 34.8, 39.0, 60.8 and 62.2, the result is in good agreement with the same angle of Cavani's report [28].

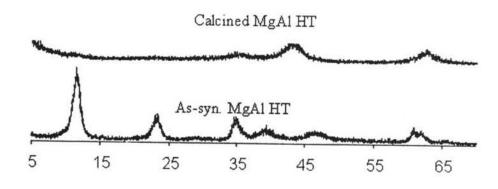


Figure 4.2 Diffractograms of as-syn. and calcined hydrotalcite.

After the hydrotalcite was calcined at 450° C, it was converted to Mg(Al)O phase, this revealed from the diffraction peaks which were shifted. The diffraction peaks were observed at $2\theta = 35.7$, 43.3 and 62.8. The unit cell dimension of cubic system is calculated to be 4.23 Å. This value is the same as report previously [28].

For the MgO catalyst, synthesized by precipitation method, the XRD pattern, illustrated in Figure 4.3, showed the presence of MgO at $2\theta = 37.0$, 42.9 and 62.4 (file 45-0946, JCPDS-ICDD) [29].

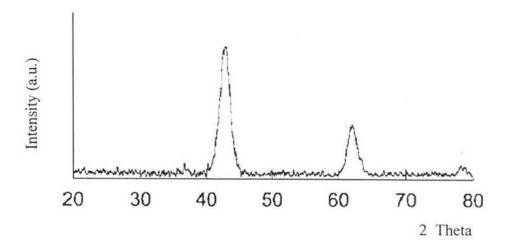


Figure 4.3 XRD pattern of MgO

4.1.2 X-ray fluorescence spectrometry (XRF)

To determine the amount of metal loaded on the mordenite either using ionexchange method or impregnation, the catalysts were analyzed by XRF at the Scientific and Technology Research Equipment Center, Chulalongkorn University, The results are shown in Table 4.2.

Table 4.1 Amounts of metal in catalysts

catalyst	%wt	Ion exchange method	Impregnation method*	
Pt-M	Pt 0.95		1	
Pd-M	Pd	1.12	1	

^{*} The amount is as loaded in the catalyst.

The XRF results showed that for the ion-exchange method, Pt-M and Pd-M catalysts contained metal content of 0.95% and 1.12%, respectively. The value of Pd is quite close to Pt content so it is reasonable that the activity of these catalysts prepared by both methods can be compared.

4.1.3 Nitrogen adsorption, Brunauer – Emmett-Teller method (BET)

All metal-loaded catalysts were characterized for their surface area, pore volume and average pore diameter by nitrogen adsorption technique. The values of the catalysts with metal loaded by ion exchange and impregnation methods are given in Table 4.2.

Table 4.2 Surface area, pore volume and average pores diameter of catalysts

Catalysts	Surface area	Total pore volume	Average pore	
	(m ² /g)	(cm ³ /g)	diameter (nm)	
НМ	551	0.35	2.56	
Impregnation				
1%Pt/HM	550	0.32	2.79	
1%Pd/HM	544	0.34	2.81	
0.5%Pt1%Ga/HM	536	0.30	2.22	
0.5%Pt2%Ga/HM	531	0.29	2.22	
1%Pt0.5%Ga/HM	525	0.35	2.66	
1%Pt1%Ga/HM	531	0.34	2.57	
1%Pt1%Zn/HM	530	0.36	2.73	
1%Pt2%Zn/HM	510	0.30	2.67	
0.5%Pt1%Ga/KCl-M	530	0.27	2.23	
0.5%Pt1%Ga/K ₂ CO ₃ -M	535	0.30	2.25	
1%Pt1%Ga1%K/HM	527	0.32	2.65	
1%Pt1%Ga0.5%K/HM	532	0.32	2.74	
Ion-exchange				
Pt-M	552	0.35	2.78	
Pd-M	547	0.33	2.80	
KCI-M	545	0.34	2.80	
K ₂ CO ₃ -M	550	0.35	2.79	

All of catalysts of this work had very large surface area (>500 m²/g) when compared with other reports (<360 m²/g) [27]. For the catalysts prepared by loading metal on the mordenite, the surface areas of these catalysts were slightly decreased. This revealed that metal oxide deposition into the mordenite pores might occur. In addition, it was found that the total pore volumes of catalysts decreased when the content of metal increased. Total pore volume of all catalysts was 0.3 cm³/g. These values were quite close to previous reported [27].

4.1.4 Fourier-transform infrared spectroscopy (FT-IR)

The metal-loaded mordenite catalysts were characterized by FT-IR. The FT-IR spectra of Pt-M and Pt/HM in which Pt was introduced by ion-exchange and impregnation method, respectively are shown in Figures 4.4. The spectra of the Pd catalysts were also shown in the same figure.

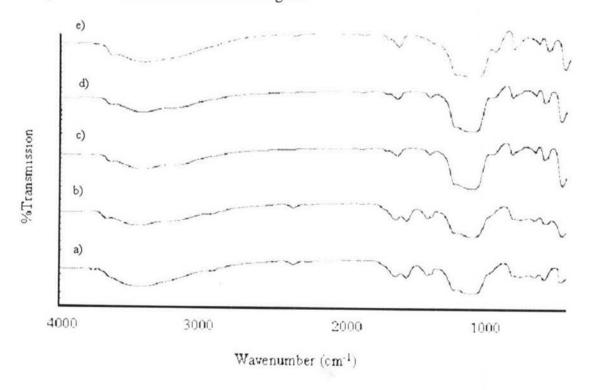


Figure 4.4 FT-IR spectra of the metal-loaded mordenite catalysts by ion-exchange and impregnation methods (a) 0.95%Pt-M, (b) 1%Pt/HM, (c) 1.12%Pd-M, (d) 1%Pd/HM and (e) HM.

The spectra of the metal loaded mordenite displayed characteristic absorption bands of mordenite, O-H stretching at 3642 cm⁻¹, O-H bending at 1397 cm⁻¹ and Si-O stretching at 1073 cm⁻¹ [30].

The FT-IR spectra of HM in Figure 4.4 (e) showed characteristic adsorption bands of mordenite that correspond with the previous report of Maiden [30]. The assignment is summarized in Table 4.5.

Table 4.3 The assignment for FT-IR spectra of mordenite catalysts

Wave number (cm ⁻¹)	Assignment	
650	Al-O (in alternating SiO ₄ and AlO ₄)	
785-775	Al-O symmetric stretching	
1081-1059	Si-O symmetric stretching	
1397	OH bending	
3600-3300	OH stretching of Si-OH	

IR absorption at about 650 cm⁻¹ is resulted from stretching of AlO₄ tetrahedral of zeolite framework. Peaks around 785-775 cm⁻¹ and 1081-1059 cm⁻¹ are Al-O and Si-O symmetric stretching. The spectra also show evidence of adsorbed water, the OH bending from adsorbed water in pore of mordenite is observed at around 1397 cm⁻¹. Absorption in range 3600-3300 cm⁻¹ can be ascribed to the OH stretching vibration of the hydroxyl groups attached to Si in elite framework.

FT-IR spectra of mono- and bimetallic mordenite catalysts prepared by impregnation method are shown in Figure 4.5.

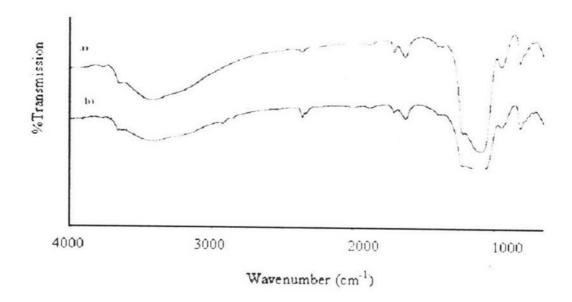


Figure 4.5 FT-IR spectra of mono- and bimetallic mordenite catalysts prepared by impregnation method. 1%Pt/HM and b) 1%Pt1%Ga/HM.

Figures 4.5 (a) and 4.5 (b) show FT-IR spectra of 1%Pt/HM and 1%Pt1%Ga/HM, respectively. The characteristic absorption peaks of mordenite are O-H stretching at 3642 cm⁻¹, O-H bending at 1397 cm⁻¹ and Si-O stretching at 1073 cm⁻¹ [31]. It can be seen that the Si-O stretching vibration shifted to higher frequency when coordinated to metal ion [29].

4.1.5 Acidity

 NH_3 -TPD technique was used to measure the acidic characteristic of catalysts. The ammonia fragment ion m/z = 16 selected to characterize ammonia is displayed in Table 4.3. All the samples showed two peaks corresponding to weak and medium-strong acidities [32].

Table 4.4 Acidity of mordenite catalysts

Catalyst	Total acidity	Weak acidity	Td	Strong acidity	Td
	$(mmol\ NH_3\ g_{cat}^{-1})$	(mmol NH ₃ g _{cat} ⁻¹)	(°C)	(mmol NH ₃ g _{cat} ⁻¹)	(°C)
НМ	1.45	0.71	170	0.74	350
0.95%Pt-M	1.06	0.48	170	0.58	400
1.12%Pd-M	1.27	0.62	168	0.65	400
1%Pt/HM	1.17	0.43	169	0.74	400
1%Pd/HM	1.30	0.68	168	0.62	400
1%Pt1%Ga/HM	1.32	0.72	169	0.60	399
1%Pt1%Ga/KM	0.84	0.40	170	0.44	398
0.5%Pt1%Zn/HM	1.13	0.48	175	0.30	450
0.5%Pt2%Zn/HM	0.74	0.54	175	0.20	450

Two weight losses occurring at two different temperature ranges may be due to the desorption of adsorbed ammonia on weak and strong acid sites. The first weight loss occurs from metal-loaded mordenite catalysts in the temperature range of 150-200°C, this can be due to the Brønsted acid site. The second weight loss at 350°-400°C is resulted from desorption from the Lewis acid sites [33, 34].

For the monometallic catalysts, after loading metal by ion-exchange (0.95%Pt-M and 1.12%Pd-M), their acidity were increased because of an increase in Lewis acid from metals. The Pd catalysts have higher acidity than Pt catalysts due to higher Pd amount, determined from XRF.

In this work, the aim of loading K onto the catalyst is to lower the catalyst acidity. In the study, it was found that the presence of K ion in 1%Pt1%Ga/KM in fact lowered the catalyst acidity as analyzed by NH₃-TPD.

TPD profiles compared between ion-exchange catalysts with mordenite support are shown in Figure 4.6.

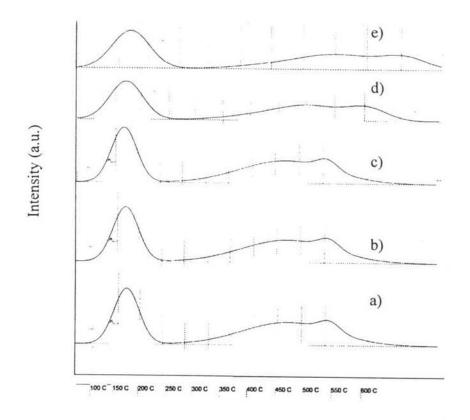


Figure 4.6 TPD profiles of catalysts a)HM, b) 0.95%Pt-M, c) 1%Pt/HM, d)1.12%Pd-M and e)1%Pd/HM.

The NH₃-TPD profile in Figure 4.6 (a)-(e) showed desorption temperature of mordenite support at two regions. The first one at low temperature (170-200°C) associates with Brønsted acid site of zeolite. The second one at high temperature (400-550°C) associates with Lewis acid site. Desorption temperature indicates acid strength of acid site, in this case Lewis acid site is stronger than Brønsted acid site [35].

To understand the effect of potassium loading on the acidity of the catalyst, NH₃-TPD of the 1%Pt1%Ga/HM and 1%Pt1%Ga/K-M catalysts were compared as shown in Figures 4.7 (a) and (b).

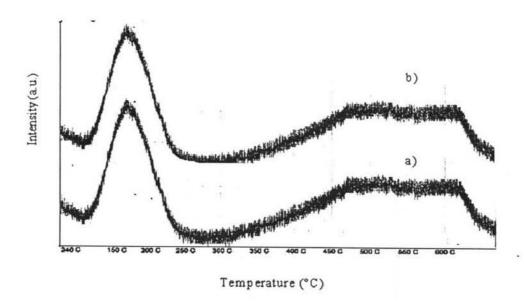


Figure 4.7 TPD profiles of catalysts. a) 1%Pt1%Ga/HM and b) 1%Pt1%Ga/KM.

The results showed that the acidity of 1%Pt1%Ga/HM is higher than that of 1%Pt1%Ga/KM. This revealed that potassium can decrease the catalyst's acidity.

4.2 Catalytic activity

Various effects influencing the catalytic activity including type of metal, metal loading method, mono- and bimetallic catalysts and time on stream were investigated.

In previous literature, Villegas *et al.* reported that skeleton isomerization of *n*-butane over Pt/mordenite catalyst gave the highest conversion at temperature of 400°C [36]. Therefore, for this work, temperature of 400°C was chosen for all experiments to compare activity of all the prepared catalysts. The results were described in details below.

4.2.1 Effect of Pt and Pd metal

The effects of metal, Pd and Pt, on both conversion and products selectivity of *n*-hexane conversion were investigated. The result is shown in Figure 4.8.

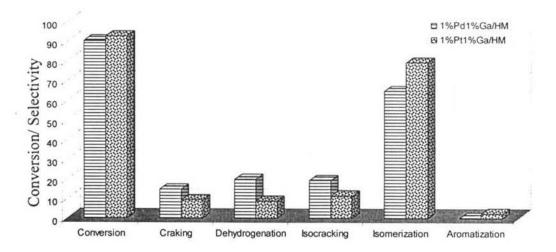


Figure 4.8 Effect of Pd and Pt metal on conversion and products selectivity. Condition: N_2 flow rate 10 ml/min, WHSV 1 h^{-1} , reaction temperature 400°C, 1.0 cm³/min n-hexane feeding rate.

From the comparison of catalytic conversion between Pt and Pd containing catalysts, the experimental results reveal that both metals have comparable activity. However, for the product distribution, it was found that Pd gave higher selectivity to olefins while Pt gave higher selectivity to isomerized products and aromatics (BTX). In the literature it was reported that Pt is more active for hydroisomerization (36% selectivity) reaction [37]. This work presented high activity of isomerization than other works because isomerization products were much more than 70%.

On the catalyst, alkane can be dehydrogenated on a metallic site to the corresponding alkene. The alkene is activated on a protonic site to form a carbocation, which is an intermediate in the isomerization. The carbocation after rearranged to produce 2-methylpentane (2MP) and to 3-methylpentane (3MP) by 1,2-alkylhydrided shift reaction (figure 2.2). The C4-isomers could be produced by the rearrangement of

cyclopropane ring intermediate to obtain 2,2-dimethylbutane (22DMB) and 2,3-dimethylbutane (23DMB) [33].

In this work, methylpentane and dimethylbutane were determined by GC-MS technique (see appendix C).

4.2.2 Effect of metal loading method on mordenite catalysts

The effect of different metal loading methods, ion exchange and impregnation methods, on conversion and products selectivity of *n*-hexane conversion was investigated.

For this experiment, the following catalysts were compared.

1%Pd/HM and 1%Pt/HM, impregnation method 1.12%Pd-M and 0.95%Pt-M, ion-exchange method.

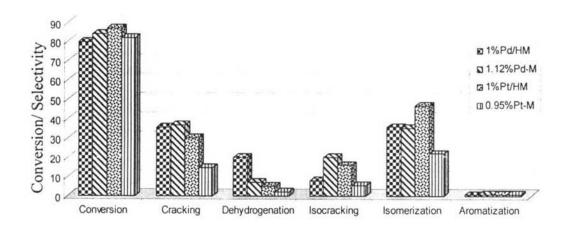


Figure 4.9 Effect of metal loading methods on conversion and products selectivity. Reaction conditions: 450°C and 1.0 cm³/min *n*-hexane feeding rate.

From experimental results, the order of the catalytic conversion is found to be 1%Pt/HM > 0.95%Pt-M. This is due to the higher Pt content in the 1%Pt/HM catalyst. Meanwhile, for the Pd catalysts, the activity order is 1.12%Pd/HM > 1%Pd-M. The same explanation is given, the higher amount of Pd. The metal contents of these catalysts are shown in Table 4.2.

In summary, the catalytic conversion is associated with the amount of metal in the catalysts. For the catalyst having the same amount of metal, the results showed that Pt containing catalysts gave higher %conversion than the Pd containing catalysts. 1%Pt/HM Catalyst was selected for further studies. So, the catalytic activities of metal loading catalyst prepared from ion-exchange and impregnation methods were not different.

4.2.3 Effect of bimetallic catalysts

The effects of bimetallic catalysts compared between 0.5%Pt1%Ga/HM, 0.5%Pt2%Ga/HM and 1%Pt1%Ga/HM on conversion and products selectivity of n-hexane conversion were investigated at the same reaction conditions: 400°C and 1.0 cm³/min n-hexane feeding rate. The results are shown in Figure 4.10.

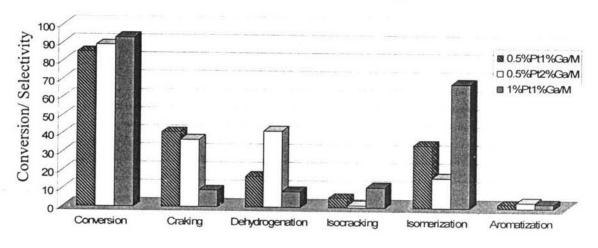


Figure 4.10 Effect of bimetallic catalysts on conversion and products selectivity.

The results showed that when the amounts of metal (Pt and Ga) were increased, the *n*-hexane conversion was increased.

Dehydrogenation and aromatics products were increased with an increase amount of Ga. This reveals that Ga metal increases the formation of unsaturated hydrocarbons. It is generally agreed that zeolite Brønsted acid sites catalyze cracking, oligomerization and cyclization reaction step and that Ga active sites are responsible for

dehydrogenation reaction of feed alkanes and intermediate products of the aromatization reaction steps [38].

When the amount of Pt was increased, isomerization products increased as shown in the comparison between 0.5%Pt1%Ga/HM and 1%Pt1%Ga/HM.

The isocracking product is also increased at higher amount of Pt, similar to the isomerized products.

In this work, Zn was also investigated and compared with Ga. The effect of bimetallic Pt and Zn containing catalysts on conversion and product selectivity is shown in Figure 4.11.

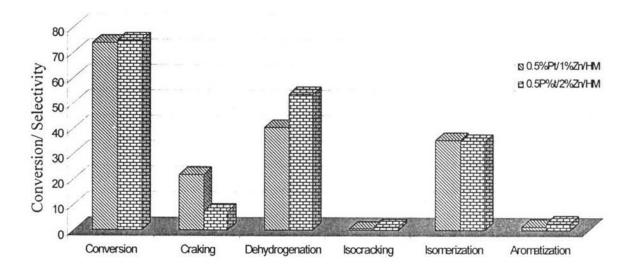


Figure 4.11 Effect of bimetallic Pt/Zn catalysts on conversion and products selectivity.

The reaction conditions are N_2 flow rate 10 ml/min, WHSV 1 h⁻¹, reaction temperature 400°C, 1.0 cm³/min *n*-hexane feeding rate.

The reaction conditions are N_2 flow rate 10 ml/min, WHSV 1 h⁻¹, reaction temperature 400°C, 1.0 cm³/min *n*-hexane feeding rate.

The results show that at 400°C the *n*-hexane conversions of 0.5%Pt1%Zn/HM and 0.5%Pt2%Zn/HM is slightly different.

Selectivity to olefins products is increased when increasing amount of Zn, this result is in good agreement with previous report which indicated that the Pt-Zn zeolite gave high olefin products for propane dehydrogenation [39].

4.2.4 Effect of potassium loading

Effect of potassium loading was studied and the results are shown in Figure 4.12.

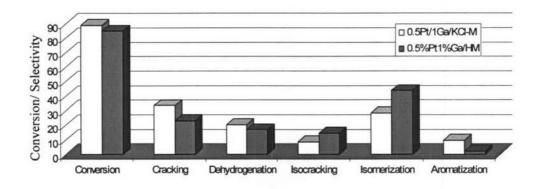


Figure 4.12 Effect of potassium salts on conversion and products selectivity of *n*-hexane conversion.

The result shows that 0.5%Pt/1%Ga/KCl-M gave higher %conversion than 0.5%Pt1%Ga/HM. Addition of KCl led to the increase in the selectivity of olefins and BTX formation accompanied with the decrease in the selectivity of isomerization. It was explained in the literature that KCl inhibits hydrocracking and hydroisomerization because the Brønsted acid site were substituted by K salt (hydrogenation sites were decrease) [16]. The result obtained from this work is in good agreement with that reported on the influence of alkali ions on the catalytic activity and selectivity of different Pt/zeolite catalysts. Potassium improves catalytic activity and isomerization activity. It modifies electronic property of the catalyst or promotes the activity of Pt.

Next, different kinds of potassium salt (KCl and K₂CO₃) were compared. The result is shown in Figure 4.13.

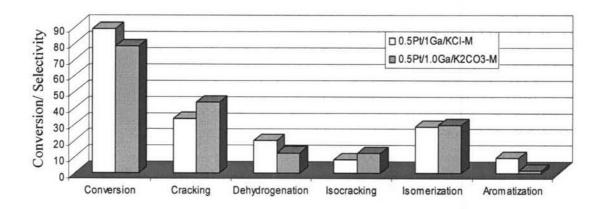


Figure 4.13 Effect of different potassium salts on conversion and products selectivity of *n*-hexane conversion.

The result shows that KCl gave higher %conversion and dehydrogenation product than K₂CO₃. Because of high electronegativity chlorine can increase positive charge of Pt particle, so the activity of metal site is increased [16].

4.2.5 Effect of time on stream

The effects of time on stream on conversion and products selectivity of n-hexane conversion were investigated. The catalysts chosen for this study are 0.5%Pt1%Ga/M catalysts. The reaction conditions are: 450°C and 1.0 cm³/min n-hexane feeding rate. The results are shown in Table 4.5 and Figures 4.14.

Table 4.5 Effect of time on stream of *n*-hexane conversion by 0.5%Pt1%Ga/HM.

Time (h)	%Conversion
3	85
5	77
8	77

The results showed that for both catalysts tested the conversion of *n*-hexane slightly decreased over time of reaction. So, the catalyst was quite stable over 8 h time period of testing.

In order to the change in product selectivity over time on stream, the products obtained for the case of using 0.5%Pt1%Ga/HM catalyst were summarized in Figure 4.14.

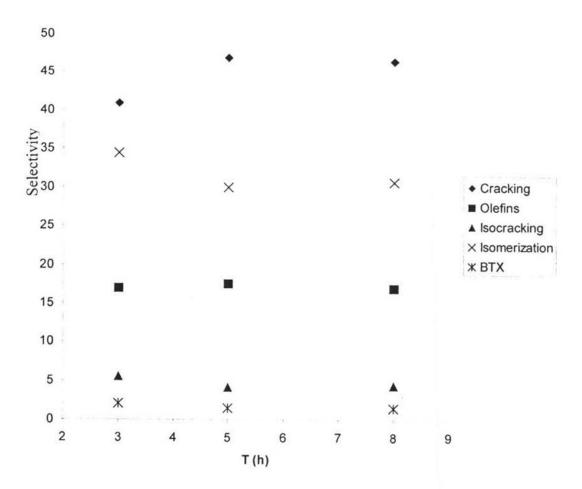


Figure 4.14 Effect of time on stream on product selectivity of *n*-hexane conversion using 0.5%Pt1%Ga/M catalyst.

4.2.6 Effect of admixture

In this work, n-hexane conversion was studied with various types of mixtures.

- (a) Metal oxide and metal mordenite mixtures
- (b) Zeolite and metal mordenite mixtures
- (c) Clay and metal mordenite mixtures
- a. Metal oxide and metal mordenite mixtures

Each metal oxide: Ga/Al₂O₃, ZrO₂ and MgO were mixed with 1%Pt1%Ga/HM in 1:1 weight ratio. The catalytic activities and product distribution were shown in Figure 4.15.

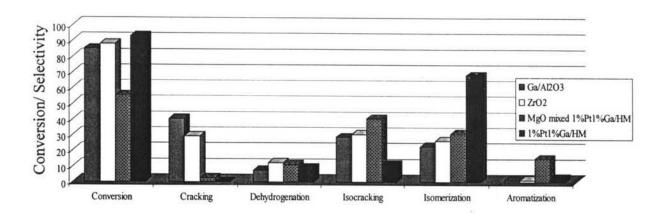


Figure 4.15 Conversion and products selectivity of *n*-hexane conversion using metal oxide and metal mordenite mixtures.

Condition: N_2 flow rate 10 ml/min, WHSV 1 h^{-1} , reaction temperature 400°C, 1.0 cm³/min n-hexane feeding rate.

The results showed that all mixed catalysts gave lower conversion than 1%Pt1%Ga/HM because the mixed catalysts had fewer metallic sites (Pt). For the mixture containing other metallic sites such as Ga, Zr and Mg had lower conversion when comparing to catalyst that contained Pt site because dehydrogenation step was favored by Pt site [37].

In the literature, Mao reported the activity of the catalyst prepared from mixing Ga (18.8% by weight) with ZSM-5 catalyst for butane reaction. The Ga species increased aromatic products of the hexane conversion. Cracking products increased (25.2%) because total acidity of the catalyst increased [39]. For this work, Ga/Al₂O₃ mixture increased cracking product due to its Brønsted acid site in catalyst. The cracking product increased (39.2%) by hydrogenation of intermediate carbocation. Aromatic products were not clearly changed by Ga species when compared with the previous report [40]. So, Ga/Al₂O₃ is not appropriate for mixture with mordenite catalyst because it gave high cracking products and could not improve aromatic selectivity.

As ZrO_2 and metal mordenite mixture, MgO and metal mordenite mixture, the results showed order of n-hexane conversion is MgO < ZrO_2 . This is due to the acidity of the catalyst. MgO is more basic than ZrO_2 , the percentage of conversion decreased

with the decreasing of acidity. ZrO₂ has weak basicity but exhibits bifunctional acidbase properties [40]. MgO favors aromatization because the dehydrogenated species generated by the Pt particles are stabilized by basic oxide ion, which is in good agreement with previous work [41].

b. Zeolite and metal mordenite mixture

MCM-41 is the zeolite chosen for this study, it was mixed with 1%Pt1%Ga/HM by physical method. The results were shown in Figure 4.16.

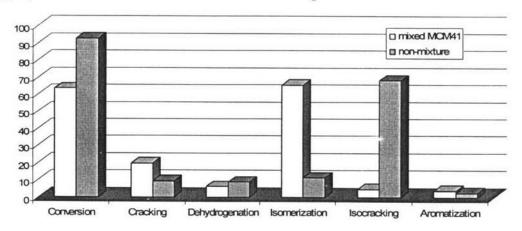


Figure 4.16 Conversion and products selectivity of *n*-hexane conversion using MCM-41 and metal mordenite mixture.

Condition: N_2 flow rate 10 ml/min, WHSV 1 h^{-1} , reaction temperature 400°C, 1.0 cm³/min n-hexane feeding rate.

Catalytic conversion of mixed MCM-41(63.5%) < non-mixture 1%Pt1%Ga/HM (92.7%) because metal was decreased by MCM-41 diluent.

MCM-41 mixture improved aromatization reaction by pore opening of zeolite. Because of MCM-41 (5-membered rings) had smaller pore than mordenite (12-membered rings), so MCM-41 increased effect of product selectivity. The main products are isomerized products. However, aromatics selectivity was still low when comparing with MCM-41 catalyst of report previously [42]. So, the MCM-41 mixture is not appropriate for aromatics formation.

c. Clay and metal mordenite mixture

The influence of clay mixture on the properties and performance of platinum-containing catalyst for *n*-hexane conversion was studied. Hydrotalcite, bentonite and potassium containing-bentonite (prepared by ion-exchange) were chosen for mixed with 1%Pt1%Ga/HM by physical method. The result was shown in Figure 4.17.

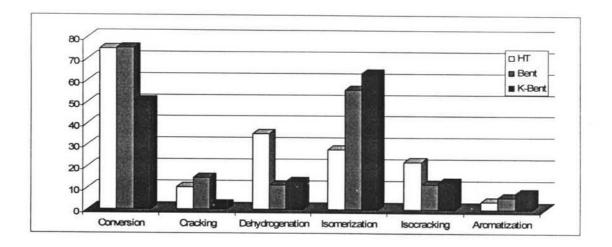


Figure 4.17 Conversion and products selectivity of *n*-hexane conversion using bentonite and metal mordenite mixture.

Condition: N_2 flow rate 10 ml/min, WHSV 1 h^{-1} , reaction temperature 400°C, 1.0 cm³/min n-hexane feeding rate.

The results in Figure 4.17, reported the conversion 1%Pt1%Ga/HM mixed with HT ~Bent > K-Bent. The main product of HT mixture is dehydrogenation due to basic function of HT. So, hydrocracking decreased and dehydrogenation increased.

Catalytic selectivity of K-Bent mixture increased aromatization product due to effect of alkali-base. The total acidity of mixture Bent (2.3 mmol $NH_3g_{cat}^{-1}$) > K-Bent (2.0 mmol $NH_3g_{cat}^{-1}$). Isomerization activity of Bent and K-Bent mixture catalysts were higher than 1%Pt1%Ga/HM catalyst because Bent and K-Bent catalysts had more acidity than mordenite material.

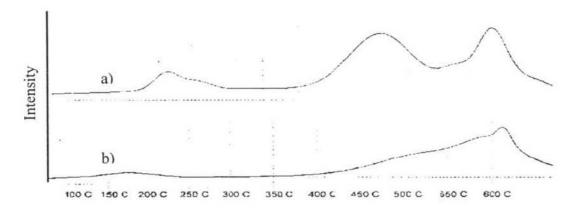


Figure 4.18 TPD profiles of mixture a) Bent and b) K-Bent.

TPD profile of bentonite in Figures 4.18 showed the acidity of Brønsted acid site of Bent and K-Bent which indicated by desorption of NH₃ at 150-300°C and Lewis acid site at 400-700°C. However, the total acidity of K-Bent was lower than the total acidity of Bent mixture because alkali metal decreased total acidity of the catalyst.

4.2.7 Regeneration of catalysts

The activities of fresh and regenerated catalysts after first and second time use were compared. The results are shown in Figure 4.19.

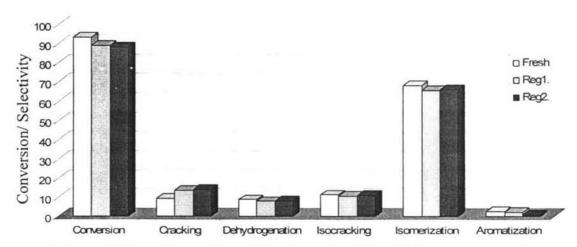


Figure 4.19 Conversion and product selectivity of *n*-hexane conversion,

compared between the fresh and regenerated catalysts.

Condition: N_2 flow rate 10 ml/min, WHSV 1 h^{-1} , reaction temperature 400°C, 1.0 cm³/min n-hexane feeding rate

It was shown that the selectivity to isomerization and aromatization for the regenerated catalyst decreased with the number of usage. This is due to coke formation

over metal site. However, it is seen that the catalyst can be regenerated and still shows some activity. The formation of coke in the spent catalyst before regeneration is revealed by TG curve (shown in Figure 4.20). In the TG curve, the loss of water in catalyst pores occurs at 100°C. At 800-900°C, coke is decomposed (3.83 wt%).

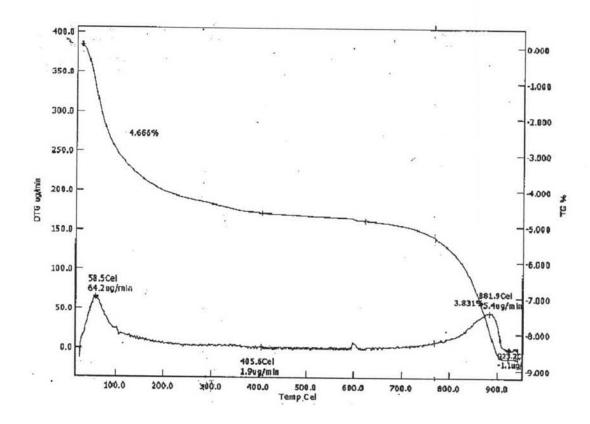


Figure 4.20 The thermogravimetric curve of the spent catalyst.