

Catalytic Conversion on Zeolites: Synthesis and Characterization

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Abstract

In this present work, catalytic cracking of durene over HY and H β zeolites, and their silica modified forms was examined at the temperature of 450 °C. The reaction was carried out in gas phase using a fix bed reactor. HY and H β zeolites showed high conversion because of their large pore size comparable to the kinetic diameter of durene. Silica modified HY zeolite showed a slight increase in selectivity, which is attributed to blocking of external acid sites. It is conceived that gradual increase in silica content could gradually enhance p-xylene selectivity. Similar effect could also be expected for H β zeolites. One can maximize toluene selectivity compared to p-xylene, but the latter is commercially more important than the former. This study depicts that other large pore zeolites could also be good to crack durene to p-xylene.

Keywords: Durene cracking, Zeolites, ZSM-5, H β zeolites, HY zeolites

1. Introduction

Presently, hydrocarbon cracking is widely used in a plenty of applications and research fields. The first step is the main process for the production of ethylene is the thermal cracking of hydrocarbon fuels [1]. The second step is the oxidative cracking of hydrocarbon which is an alternative process to steam cracking for production of olefins [2, 3]. The third step is the pyrolysis of hydrocarbon (e.g. low-density polyethylene) in a quartz semi continuous feed reactor with fast heating to obtain solid carbons [4-5]. In recent years, the thermal decomposition of hydrocarbon fuel in hypersonic aircrafts is also a research hotspot, because hydrocarbon fuel is required not only for the propulsive power but also as a coolant with high cooling capacity due to its endothermic pyrolysis reaction at high temperature [6–8]. But, the tradition cracking process need high temperature and cause many environmental related problems. To avoid these limitations, catalyst cracking has been employed in the industrial process using different types of catalysts like transition metals [9-10], silica [11], zeolite and modified zeolite [12], mesoporous mixed oxide catalysts [13] and metal organic frame works.

In the early 1970s, the MTG (methanol to gasoline) process was identified and patented by Mobil Oil Corporation [14–17]. But from the late 1970s, there have been plenty of literatures on the MTG process [18–22]; in which methanol is converted to gasoline over ZSM-5 derived catalysts.

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A dual bed of methanol synthesis catalyst followed by the H-ZSM-5 catalyst was used in these studies. Most of the research studies have been carried out using a continuous flow reactor. As per literatures, there has been only one study for MTG performed in a batch reactor [23]. The widely recognized mechanism for the MTG process involves conversion of methanol to dimethyl ether, which is further converted to ethene and propene. These alkenes in turn form aromatics and other gasoline-range hydrocarbons inside the zeolite pores [24].

Generally, Zeolites are crystalline aluminosilicates which possess Brønsted acidic sites located within ordered micropores of molecular dimensions. Among the other catalysts, these zeolites is considered to remain the best catalysts for the cracking of oils, due to its high thermal stability, good shape-selective properties resulting from their microcrystalline structures, and their ability to concentrate reactants inside their pores [25-26]. For instance, the H-ZSM-5 catalyst has a medium size pore system, *i.e.* pores which circumference is defined by rings consisting of 10 T-atoms. Straight channels (5.1 Å x 5.5 Å) are intersected by zigzag channels (5.3 Å x 5.6 Å), thus creating a three-dimensional network [27-20]. Various kind of zeolites like HY and H β zeolites are also widely used for various chemical reactions, not only as catalyst but it also supports due to their excellent thermal stability and shape-selective properties [28-29]. HY has been used as a catalyst in cracking reactions of palm oil [30] and soybean oil [31], and the products obtained had compositions similar to those of gasoline.

Durene (1, 2, 4, 5-tetramethylbenzene) is considered to be one of the main series of p-complexing agents, which was investigated by O'Driscoll [32] for the anionic polymerization of styrene in benzene, Durene is an electron-rich arene. For industrial applications, it is the main precursor to pyromellitic dianhydride, which is used for manufacturing curing agents, adhesives and coating materials. It is also used in the manufacture of some raw materials for engineering plastics (polyimides) and cross-linking agent for alkyd resins.

The gasoline produced by the MTG process contains durene, and its content is more than that permitted under product gasoline specifications. The durene content in gasoline is reduced by treating the heavy gasoline produced in MTG, in the Heavy Gasoline Treatment plants prior to blending into product gasoline. The cracking pattern and the catalyst employed are not clearly known. Moreover the catalytic transformation of durene into any other value added products have not been attempted to the best of our knowledge. Our initial attempt on catalytic conversion of durene into p-xylene was published [33]. ZSM-5 was used as the catalyst, but since the pore diameter of it was not adequate compared to the kinetic diameter of durene, the conversion of durene was lower than 30%. In this present study large pore HY and H β zeolites and their silica modified form such as, SiO₂ (30%)/HY and SiO₂ (30%)/H β zeolites were tested for durene transformation. SiO₂ (10%)/ZSM-5 was also examined and its activity and selectivity are compared. The results were much satisfactory and are discussed. Additional characterizations of the catalysts not discussed in the previous paper are also included.

2. Experimental

2.1. Materials

ZSM-5, HY and H β zeolite were purchased from Zeolyte International with SiO₂/Al₂O₃ ratio of 23, 2.55 and 12.5 respectively. Tetraethyl ortho silicate was purchased from Samchun Chemicals. Durene (98%) was obtained from Tokyo chemical industry, and other common chemical and solvents from Daejung Chemical, South Korea.

2.2. Characterization Techniques

Powder X-ray diffraction patterns were recorded using a Rigaku D/Max 2200+Ultima

diffractometer with Cu-K α radiation ($\lambda=0.154$ nm). The diffraction data were recorded in the 2θ range 5-50° with a step of 0.02 °/s. The nitrogen adsorption-desorption isotherms of the prepared samples were measured at -196 °C with a Belsorp mini II sorption analyzer. Prior to each adsorption measurement the samples were degassed at 150 °C under vacuum ($p<10^{-5}$ mbar) in the de-gas port. The surface area was determined from the linear part of the Brunauer-Emmett-Teller (BET) equation. The morphologies of the prepared catalysts were studied by scanning electron microscopy after gold coating using a JOEL instrument operating at 30 keV.

An in-house fixed bed reactor was used for the cracking of durene using ZSM-5, HY and H β zeolites in gas phase. The detailed experimental condition was depicted in our previous published paper [33]. The silica modified ZSM-5, HY and H β zeolites were prepared by wet impregnation method using tetraethyl ortho silicate as silica source with different weigh percentage.

3. Results and Discussion

3.1. Textural Properties

The results of the textural properties of the zeolites used in this study are presented in **Table 1**. A common observation is decrease in surface area for all the silica modified zeolites compared to their respective parent zeolites. The reduced pore volume of silica modified zeolites is not due to filling of the pores by silica formed from tetra ethyl ortho silicate which cannot enter the pores of either ZSM-5 or Y-zeolites. So, the silica has to be formed on the external surface. It is due to decrease in the zeolite content in such zeolites.

Table 1. Textural Properties of ZSM-5, HY, H β and Silica Modified Zeolites

Sample	V_m ($\text{cm}^3(\text{STP}) \text{g}^{-1}$)	Surface area ($\text{m}^2 \text{g}^{-1}$)	Pore Volume ($\text{cm}^3 \text{g}^{-1}$)	Pore Diameter (nm)
ZSM – 5	129.84	565.13	0.3105	2.1976
ZSM5 / 10% SiO ₂	88.13	383.62	0.1802	1.8786
Y – Zeolite	178.83	778.35	0.3461	1.7786
β – Zeolite	140.23	610.34	0.7221	4.7324
30% SiO ₂ / β Zeolite	133.75	582.13	0.4953	3.4034

3.2. X-Ray Diffraction Studies

The XRD patterns of ZSM-5 and 10% SiO₂/ZSM-5 are shown in Figure 1. Both the spectra showed the characteristic peaks of ZSM-5 ($2\theta = 24.47$ and 45.67) reported in the literature [34-36]. There was a slight decrease in intensity for the peaks of 10% SiO₂/ZSM-5 compared to that of the parent ZSM-5 due to false scattering of diffracted rays of ZSM-5 by the amorphous silica particles. Decrease in the intensity was very much clear in the original spectrum which is not shown.

The XRD patterns of Y-zeolite and 30% SiO₂-Y zeolite are shown in Figure 2. The HY-containing catalysts showed many narrow peaks at 2θ values of 10.3°, 12.2°, 15.6°, 18.4°, 20.3°, 23.7°, 27.0°, 31.8°, 34.2°, 38.4° and 54.6° which are typical of zeolite faujasite [37]. As expected, the spectrum of 30% SiO₂-Y zeolite also showed the characteristic peaks of Y-zeolite, but the intensity of the peaks was slightly lower than that of the parent zeolite similar to that of the SiO₂ modified ZSM-5 zeolite. Due to amorphous silica the base line of the spectrum of 30% SiO₂-Y zeolite was slightly raised up, but this observation was not noticed in modified ZSM-5 zeolite, as it was only 10% modified.

The XRD patterns of β -zeolite and 30% SiO_2 - β zeolite are shown in Figure 3. Both the spectra showed the characteristic peaks of β zeolite reported in the literature [38]. Compared to the parent β zeolite, silica modified β zeolite showed a decrease in intensity of the peaks similar to that of modified ZSM-5 and Y-zeolites.

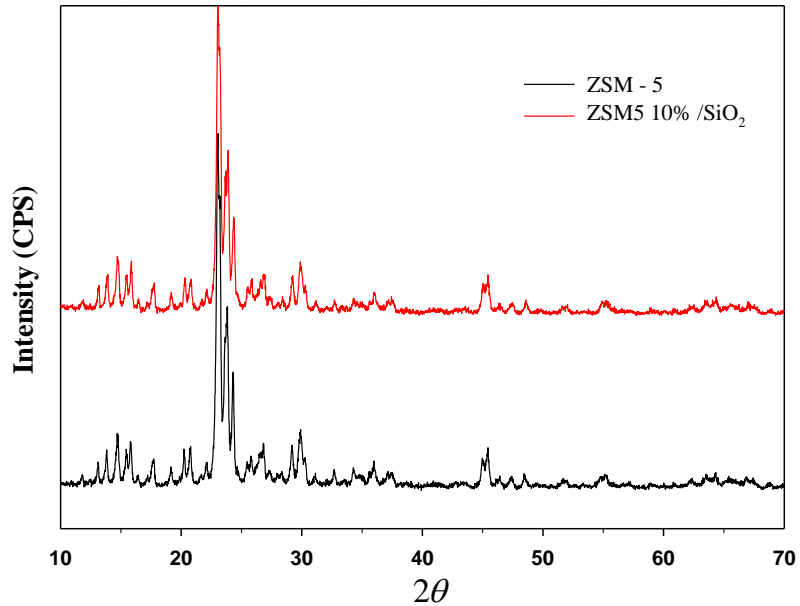


Figure 1. XRD Patterns of ZSM-5 and 10% SiO_2 /ZSM-5

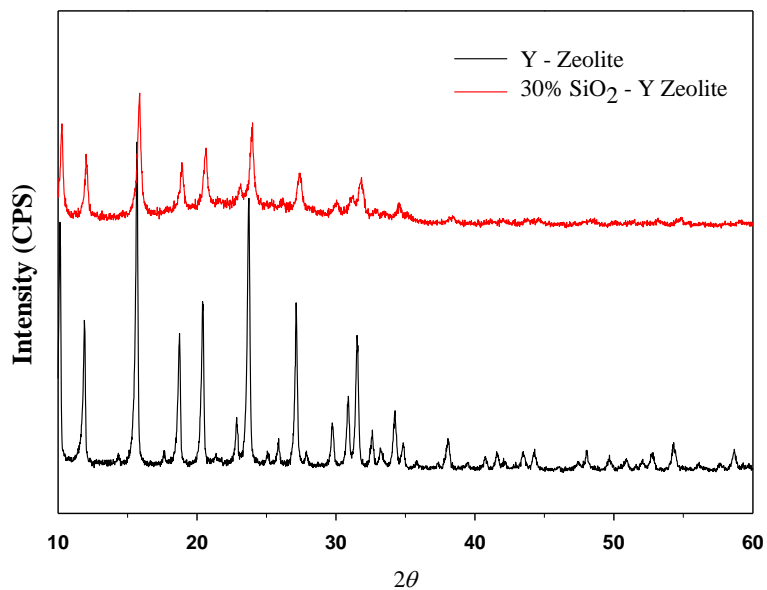


Figure 2. XRD Patterns of Y-Zeolite and 30% SiO_2 -Y Zeolite

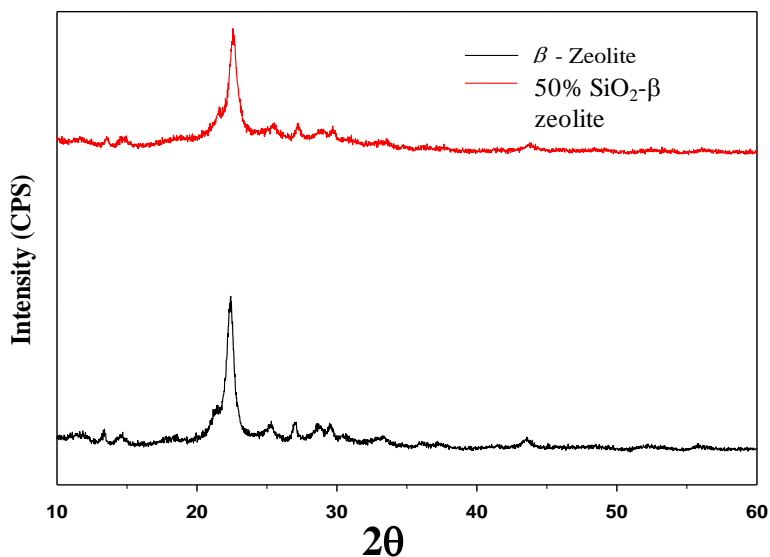


Figure 3. XRD Patterns of β -Zeolite and 50% SiO₂- β Zeolite

3.3. Scanning Electron Microscopy

The SEM images of ZSM-5, Y and β -zeolites and their silica modified forms are shown in Figure 4. The ZSM-5 image showed tiny planar crystals which were very much aggregated. The image of silica modified ZSM-5 showed particles of smaller aggregates than the ZSM-5 zeolite. The SEM images of Y-zeolites and β zeolites are shown in Figure 4(c) and (d) respectively. Both the images revealed particles of irregular morphologies.

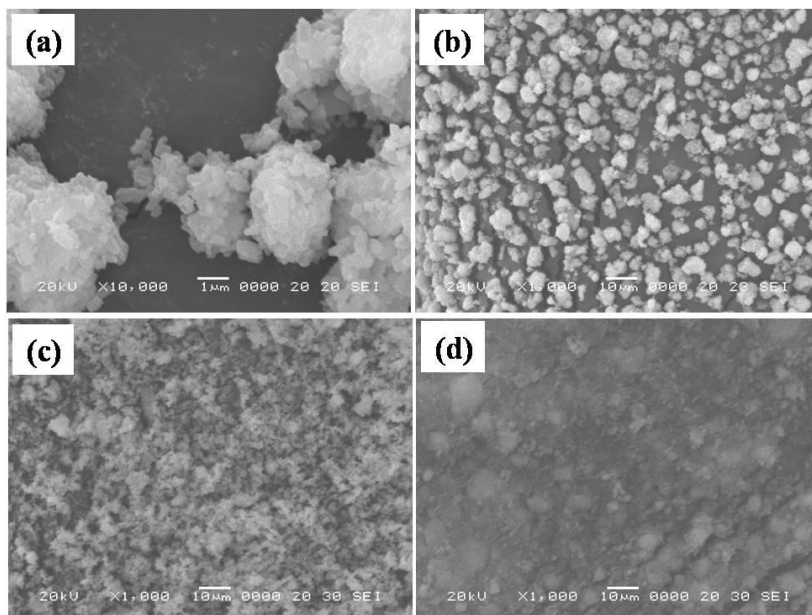


Figure 4. SEM Images of (a) ZSM-5, (b) SiO₂/ZSM-5, (c) Y-Zeolite and (d) β -Zeolite

3.4. Effect of Silica Loading

As the external surface acid sites, play a major role in reducing selectivity to p-xylene,

in this study it was attempted to block them through silica impregnation. So, 10, 20, 30 and 40% silica impregnated HZSM-5 catalysts were examined for p-selectivity. The results are illustrated in Figure 5. Generally conversion is expected to decrease with the increase in silica loading, but such trend was not followed up to 20% silica loading. At 30% silica loading significant drop in conversion was noted. P-Xylene selectivity is expected to increase with the increase in silica loading, but it also showed unexpected results. Non-uniform deposition of silica might be the cause for such results.

Cracking of durene and the products distribution within the pores of large pore zeolite or on the external surface of ZSM-5 are summarized in the reaction in scheme 1. Cracking of durene initially yields toluene and trimethyl benzene. It is an example of trans methylation from durene to benzene. The same reaction between trimethyl benzene and toluene yields p-Xylene. Trimethyl benzene was not observed in the reaction; hence it might be highly susceptible to demethylation. Durene is non-polar whereas trimethyl benzene is polar, and hence the later can be better retained in the micropores of the zeolites than the former to yield p-xylene via. trans alkylation.

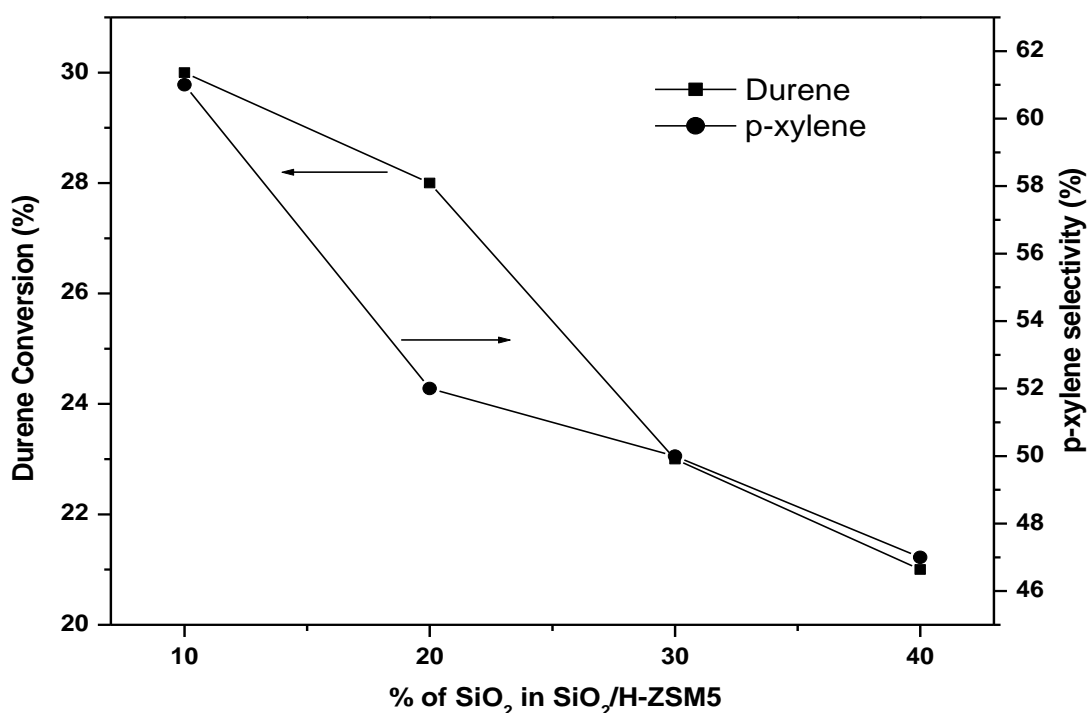
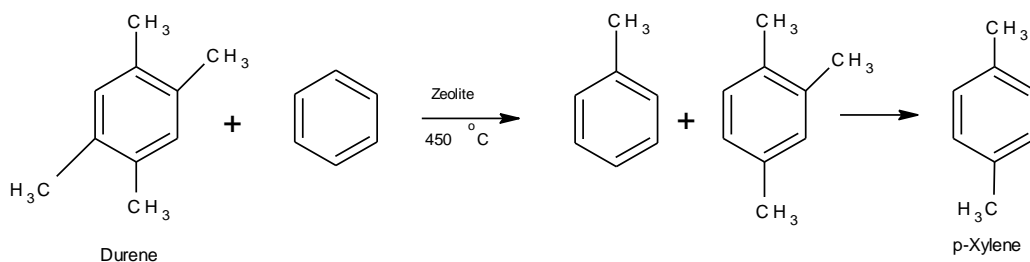


Figure 5. Effect of SiO₂ Loading of H-ZSM5 on Durene Conversion and p-xylene Selectivity (Weight of the Catalyst: 0.5g; Feed rRate: 2 mL/h; WHSV: 3.36h⁻¹, Feed Ratio: Durene: Benzene = 1:5.7; Temperature: 450 °C)



Scheme 1. Durene Cracking and the Product Distribution

3.5. Effect of Time on Stream

The effect of time on stream on durene conversion and *p*-xylene selectivity was studied over 10% SiO₂/ZSM-5, and the plot of time versus conversion and *p*-xylene selectivity is shown in Figure 6. Both the conversion and *p*-xylene selectivity rapidly decreased during the early stage of time on stream, but after 2 h both remained almost steady. It is due to partial blocking of active sites by coke on the external acid sites. Rapid fall in conversion and durene selectivity suggests that ZSM-5 zeolite is not good for the cracking of toluene, the reaction occurs only on the external acid sites not inside the pores, as the kinetic diameter of durene (7.4 Å) is higher than the pore diameter of ZSM-5 (5.4 Å).

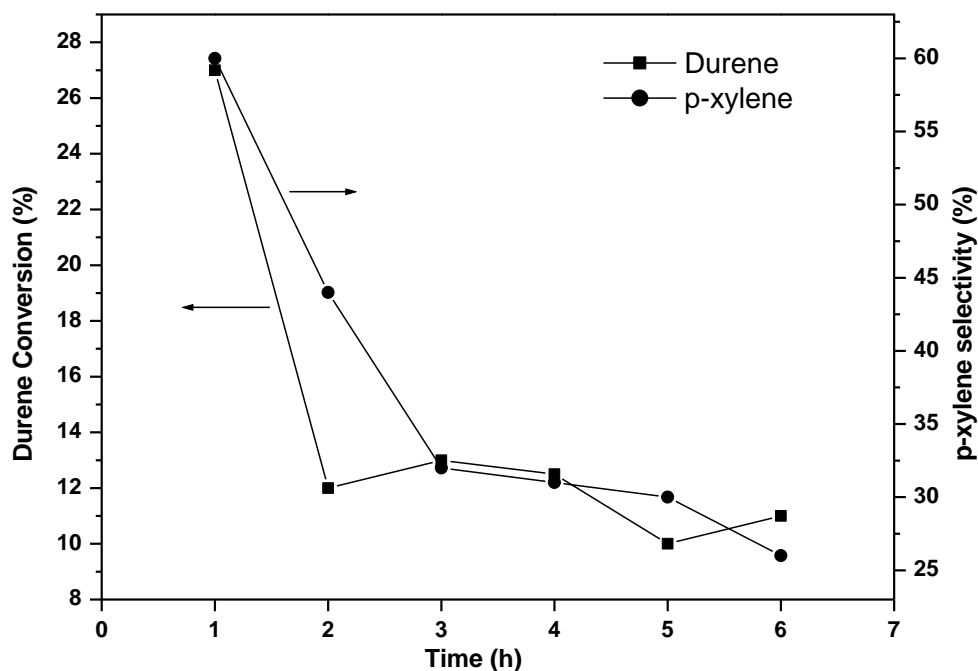


Figure 6. Effect of Time on Stream Durene Conversion and *p*-xylene selectivity (Content: 10% SiO₂/ZSM-5; Weight of the catalyst: 0.5g; Feed rate: 1 mL/h; WHSV: 1.68h⁻¹; Feed Ratio: Durene: Benzene = 1:5.7; Temperature: 450 °C)

3.6. Durene Cracking Over Large Pore Zeolites

As ZSM-5 is a medium pore zeolite with pore diameter 5.4 Å, it is verified not adequate to crack durene with kinetic diameter 7.4 Å. Hence, large pore zeolites such as HY and H β were examined. The results are presented in Table 2. The former carries super cages, whereas the latter only straight pores. Both Y and β zeolites showed very high conversion compared to ZSM-5 zeolite, as there could be constrain free diffusion of durene into the pores of both of them. Because of large pore size, toluene selectivity was higher than *p*-xylene. Toluene once formed can rapidly diffuse out, and so it gained high selectivity. High toluene selectivity is also due to high feed rate (2 mL/h), by which consecutive alkylation can be suppressed. So, lowering of feed rate can increase contact time and enhance *p*-xylene selectivity. Consecutive alkylation of toluene need not give *p*-xylene with high selectivity, as ortho xylene is also a favored isomer. For ortho substitution there might be little steric hindrance due to Van der Waals repulsion between the methyl group already present on the aromatic ring and the attacking methyl carbonium ion, but for para substitution such hindrance no longer exists. So, *o*-xylene selectivity was not much in any part of the present study. Because of large pore diameter, both the zeolites showed nearly similar level of conversion and durene selectivity.

Table 2. Durene Conversion and p-Xylene Selectivity Over γ and β Zeolites (Weight of the Catalyst: 0.5 g; Feed Rate: 2 mL/h; WHSV: 3.36h⁻¹; Durene: Benzene = 1:5.7; Temperature: 450 °C)

Sample	Durene Unconverted (%)	Durene Conversion (%)	p-xylene yield (weight %)	Toluene yield (weight %)	p-xylene Selectivity (%)
40% SiO ₂ /H β	7.46	92.54	21.05	60.34	35.45
40% SiO ₂ /HY	13.47	86.53	23.67	74.23	31.89
H β -Zeolite	5.43	94.57	19.2	49.03	26.69
HY-Zeolite	4.20	95.80	20.99	62.43	28.52

30% SiO₂/HY showed slightly lower conversion but higher selectivity than the HY zeolite. Hence the external acid sites might be partly blocked by silica. Much decrease in conversion is expected, as the zeolite content was low. So, silica might not be uniformly deposited on the zeolite. A slight increase in p-xylene selectivity over 30% SiO₂/HY compared to HY zeolite suggests isomerization of p-xylene to m-xylene could be significant over the external acid sites of the later. Over 40% SiO₂/HY zeolite both the conversion and p-xylene selectivity only slightly decreased compared to 30% SiO₂/HY substantiating our view.

3.7. Effect of Time on Stream

The effect of time on stream on durene conversion and toluene and p-xylene selectivity over 30% SiO₂/HY was studied at 450 °C and the results are illustrated in Figure 7. Both the durene conversion and p-xylene selectivity decreased with the increase in time on stream due to a gradual increase in the blocking of active sites by coke. So, periodic activation of the catalyst is required for this reaction. It is not a problem for zeolite structure, as it is thermally stable. Selective conversion of benzene to toluene could be effected with low concentration of methyl cations, but selective formation of p-xylene requires high concentration of it.

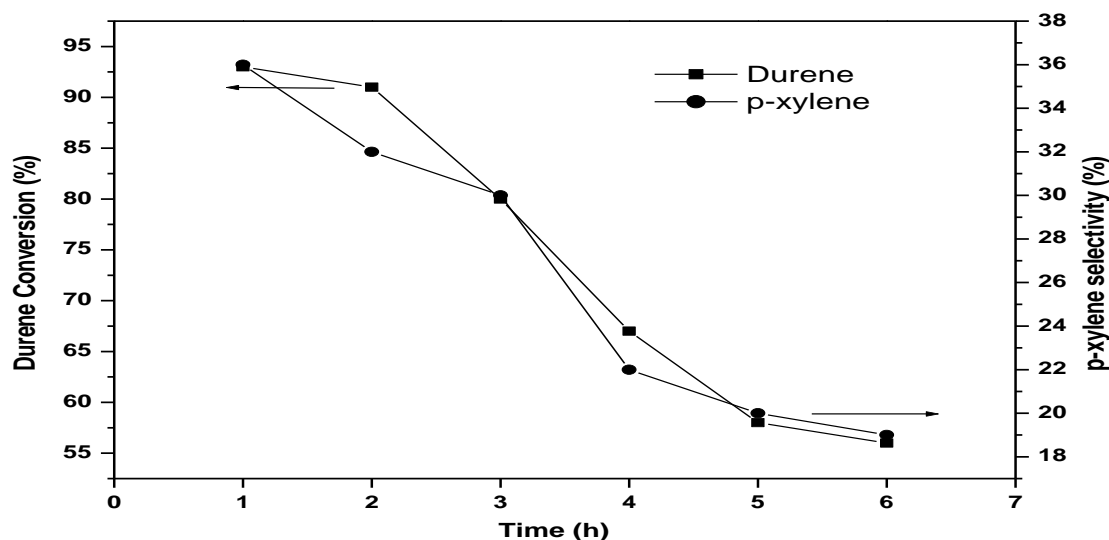


Figure 7. Effect of Time on Durene Conversion and P-Xylene Selectivity Over 30% SiO₂/HY Zeolite (Weight of the Catalyst: 0.5g; Feed Rate: 1mL/h; WHSV: 1.68h⁻¹; Feed Ratio: Durene: Benzene = 1:5.7; Temperature: 450 °C)

4. Conclusions

Cracking of durene over ZSM-5 was not good due to its high kinetic diameter compared to the pore size of the later. With this zeolite the cracking is essentially due to external acid sites and availability of defective channel intersections on the external surface. HY and H β zeolites showed high conversion because of their large pore size comparable to the kinetic diameter of durene. Though conversion was good, selectivity of *p*-xylene was not high because of rapid diffusion of toluene through the pores. Based on the results it is expected that a slight decrease in flow rate of the feed could enhance *p*-xylene selectivity. Addition of little amount of toluene to durene –benzene feed could also enhance *p*-xylene selectivity. So, use of different feed ratios of benzene and toluene could solve the problem of *p*-xylene selectivity. Silica modified HY zeolite showed a slight increase in selectivity, which is attributed to blocking of external acid sites. It is conceived that gradual increase in silica content could gradually enhance *p*-xylene selectivity. Similar effect could also be expected for H β zeolite. One can maximize toluene selectivity compared to *p*-xylene, but the latter is commercially more important than the former.

Acknowledgments

This work was supported by grants from Hanseo University and Korea CCS R&D Centre, funded by the Ministry of Education, Science and Technology of the Korean Government.

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