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**ORIGINAL RESEARCH**

# Catalytic Cracking of Polypropylene-Low Density Polyethylene Pyrolysis Oil over Hierarchical H-ZSM-5 Catalyst

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## Abstract

The conversion of plastic waste into liquid fuel has become the center of attention by researchers as a way to overcome environmental problems. According to last research, the polypropylene (PP) and low-density polyethylene (LDPE) pyrolysis oil mixture was classified into hydrocarbon range diesel which have the number of carbon atom from C12-C24. Because that, the effect of LDPE/PP pyrolysis oil over hierarchical H-ZSM-5 catalyst on hydrocarbon composition of liquid fuel oil using the catalytic cracking reactor at 200 $^{o}C$ was investigated. Hierarchical H-ZSM-5 catalyst (hH-ZSM-5) was obtained from H-ZSM-5 modified by desilication method using NaOH. Gas chromatography-mass spectrometry showed that the liquid fuel oil from the catalytic cracking of all variations consisted of aliphatic hydrocarbons with some cycloaliphatic compounds. In ratio of 250 mL/0.5 g (oil/catalyst) aromatic hydrocarbon increased from 1.69 area% to 15.58 area% over H-ZSM5 and hH-ZSM-5 respectively. The higher oil quality with shorter hydrocarbon chains in all variations was obtained over hH-ZSM-5 catalyst, namely in the range of C7-C14 or comparable to hydrocarbon range gasoline and kerosene. .

**KEYWORDS:**

PP; LDPE; ZSM-5; catalytic cracking; hydrocarbons.

## 1 | INTRODUCTION

Excessive use of plastic is one of the biggest problems today. The types of plastic that are the largest contributor to plastic waste are LDPE (Low Density Polyethylene) and PP (Polypropylene). LDPE waste accumulates as one of the largest contributors of plastic waste in Indonesia[1] [2] [3] [4]. The type of polypropylene (PP) plastic contributes as the second largest plastic waste after LDPE [5].

Catalytic cracking has the potential to convert plastic waste into alternative fuels. Catalytic cracking is one of the most popular processes used by the petroleum refining industry[4]. One of the catalysts in the petrochemical industry is ZSM-5 (Zeolite Silica Mobile-5). However, the weakness of ZSM-5 as a catalyst is its micro-sized pores[3]. Modification of ZSM-5 catalyst is required to form larger porosity, namely mesoporous (2-50 nm) to facilitate molecular diffusion. Zeolites that have both micropores and mesopores are called hierarchical zeolites. So that the H-ZSM-5 and hH-ZSM-5 (hierarchical H-ZSM-5) catalysts are obtained.

In this study, a cracking process was carried out using a mixture of PP/LDPE (25/75) % plastic oil obtained from previous research conducted by Ulfa et al. (2019). In this study, comparisons (Volume/Mass) of Oil/Catalyst were carried out, namely: (200/0.5), (250/0.5), (300/0.5), (350/0.5) and (400/0.5) for each cracking process.

The results of the XRD instrument catalyst analysis, on H-ZSM-5 showed a peak with a fairly high intensity at an angle of $2\vartheta = 7.9444°$ and Na-ZSM-5 the occurrence of angle shift at $2\vartheta = 7.8985$. The results of the SEM-EDX analysis showed the formation of Na crystals on the H-ZSM-5 catalyst and the formation of Na-ZSM-5. The results of FTIR-Pyridine spectrometry, the results of the calculation of the acid site on H-ZSM-5 were 0.074743 mmol/g and hH-ZSM-5 0.162505 mmol/g.

XRD showed that the structure of ZSM-5 was well maintained after alkali treatment, but the intensity of most of the peaks decreased slightly, indicating that alkali treatment did not damage the crystal structure of ZSM-5, but that the structure was finely modulated. The increase in acid sites increases the process of breaking/cracking of the hydrocarbon chains in plastic oils.

In the results of the cracking process, the best results were obtained for the yield percentage, namely on H-ZSM-5 in a comparison of (250/0.5) with 79.6% and on hH-ZSM-5 with a ratio of (350/0.5) with 50.8%. The results of the GC-MS analysis for the H-ZSM-5 catalyst show the best hydrocarbon range in the variation (350/0.5) which is in the C6-C22 range which is equivalent to diesel/diesel. While the results of the GC-MS analysis showed that the best results were obtained from the type of hH-ZSM-5 catalyst with a hydrocarbon range of C7-C14 or equivalent to kerosene for all variations.

## 2 | PREVIOUS RESEARCHES

1. Verboekend dan Ramı´rez/ 2011
   Design Of Hierarchical Zeolite Catalysts By Desilication
   Desilication in alkaline media has become a widely applied preparation method to adapt these modified zeolites, due to the optimal combination of efficiency and simplicity. This review presents recent developments that have broadened general understanding and made this top-down treatment highly flexible, controllable, and scalable. Design aspects of mesoporous zeolites for catalytic applications are emphasized, covering the establishment of synthesis-property-function relationships. Alkaline treatment is a key step in strategic combination with other post-synthesis modifications towards superior zeolite catalysts.

2. Sadowska et al /2017
   Desilication of highly siliceous zeolite ZSM-5 with NaOH and NaOH/tetrabuty amine hydroxide
   Desilication was carried out in a solution of NaOH and a mixture of NaOH and TBAOH (tetrabutylamine hydroxide) at temperatures of 338 and 353 K for 0.5 hours. 100 ml of solution was added to 3.0 g of zeolite. If desilication is carried out only with NaOH solution, 0.1, 0.2, and 0.5 M are used. The NaOH/TBAOH mixture has concentrations of 0.2 and 0.5 M. For the 0.2 M mixture TBAOH/(NaOH + TBAOH) ratio (denoted immediately as R) is 0.1 and 0.4, for the R solution the ratio is 0, 5 M is 0.1. After desilication, the suspension was cooled in an ice bath and filtered. At this stage, the collected filtrate samples are taken for analysis. The hierarchically structured zeolite was washed with water until the pH was neutral. Next, fourfold ion exchange

with 0.5 M NH4NO3 was carried out at 333 K for 1 h. Finally, the zeolite was filtered again, washed, and dried at room temperature. The results obtained are: The results of chemical analysis and XPS analysis show that the desilication of highly siliceous ZSM-5 from Si / Al = 164 is more effective in the surface zone than in the bulk zeolite. For the lower Si/Al zeolites = 31.6 desilication is more effective than the bulk, what is explained by the excess Al on the surface and the protection of neighboring Si atoms with negatively charged AlO4 against OH attack.

3. Groen et al/ 2008
   Mesoporous beta zeolite obtained by Desilication
   Zeolite beta crystals (Si/Al = 35) synthesized in fluoride medium were treated in 0.2 M NaOH solution for mesopore formation by selective extraction of the silicon framework. The parallel reactor-16 was used to study the effect of treatment time and temperature on the physico-chemical properties of zeolites, which were characterized by ICP-OES, XRD, N2 adsorption at 77 K, SEM, TEM, DRIFTS, and in situ ATR-IR. As a result, this stems from substantial mesoporosity and may improve transport, but negatively impacts the microstructure and acidity of the resulting samples. Consequently, base-treated beta zeolite exhibits lower catalytic activity in acid-catalyzed liquid-phase benzene alkylation than the pure microporous parent material.

4. Sartipi et al/ 2013
   Hierarchical H-ZSM-5supported cobalt for the direct synthesis of
   gasoline-range hydrocarbons from syngas: Advantages, limitations, and mechanistic insight. Mesostructures were created in H-ZSM-5 crystallites by demetalation via subsequent alkaline and acidic treatments. Desilication via alkaline treatment gives H-ZSM-5 a pore size and volume similar to amorphous SiO2 (conventional carrier), while acid treatment removes the produced extraframework aluminum and increases FTS catalyst activity. The acid-catalyzed reaction model and the induced deactivation of the zeolite acid sites confirmed that hydrocracking of primary FTS hydrocarbons on the zeolite greatly increased the selectivity towards C5-C11 (gasoline fraction). On the other hand, strong Co-zeolite interactions as revealed by TPR(H2) result in lower stabilization of coordinated Co sites (as revealed by IR-assisted CO adsorption) and in higher selectivity towards methane. The conversion reaction of n-hexane shows that the latter is due to increased activity for hydrogenation reactions and hydrocarbon hydrogenolysis at coordinatively unsaturated Co sites.

## 3 | METHOD

### 3.1 | Material and Method

In this study the main ingredient is oil from the pyrolysis of a mixture of PP and LDPE plastic waste with various ratios (75/25) %. This variation was chosen because the best ratio of mixed pyrolysis is PP: LDPE (75:25) % with the highest heating value of 46,955 MJ/Kg, density of 7.02 g/mL and viscosity 0.55 mm2/s. The results of the GC-MS analysis show that PP plastic pyrolysis oil, a mixture of PP: LDPE and LDPE belongs to the type of diesel fuel that has a C12-C24 carbon chain[2]. Commercial activated carbon, commercial catalyst Zeolite ZSM-5 Ammonium (NH4-ZSM-5) obtained from Alfa Aesar with code (ALFA45881), NaOH, NH4NO3, HNO3 and aquades.

The hierarchical H-ZSM-5 (hH-ZSM-5) catalyst preparation method was carried out according to the procedure carried out by[1]. Solid ammonium-ZSM-5 was calcined at 500 oC for 5 hours to obtain the H-ZSM-5 form. Then, the H-ZSM-5 powder was desiccated with NaOH solution of 1 M (vol. base/weight= 8.0 cm3 g-1) while stirring at 70 $^oC$ for 1 hour.

The catalytic cracking reaction was carried out in the reactor. For each test of the catalytic cracking reaction, the volume variations of the pyrolysis oil were 200, 250, 300, 350 and 400 mL and 0.5 g of H-ZSM-5 catalyst and 0.5 g of H-ZSM-5 catalyst. The catalyst of hH-ZSM-5 was put into the reactor at a reaction temperature of 200 $^oC$ for 60 minutes.

## 3.2 | Catalyst Characterization Analysis

In this study, the catalyst was analyzed by, XRD (X-Ray Diffraction) to observe the crystalline phase change in H-ZSM 5 before and after the desilication process, SEM-EDX (Scanning Electron Microscope) to observe and describe the surface of the ZSM-5 catalyst and serve as the basis for an important analytical technique for measuring the surface area of the ZSM-5 catalyst. FTIR-Pyridine (Fourier-transform infrared spectroscopy) to observe and explain the type of bond in the ZSM-5 catalyst and to function as a determination of the acidity level on the surface of the ZSM-5 and BET catalysts (Brunauer-Emmett-Teller) to observe and explain the physical adsorption of gas molecules on the surface of the ZSM-5 catalyst and serve as the basis for an important analytical technique for measuring the surface area of the ZSM-5 catalyst.

## 3.3 | Characterization of Cracked Liquid Products

For the determination of the chemical compounds of the Cracked Liquid Products and the percentage distribution of the carbon numbers of the chemical compounds, the Agilent 19091S-433 Gas Chromatography-Mass Spectrometry (GC-MS) instrument was used, with Max temperature: 325 $^{o}C$ and Nominal init pressure: 9.88 psi.

# 4 | RESULTS AND DISCUSSION

## 4.1 | Characterization Results of H-ZSM-5 and hH-ZSM-5

Fig. 1 shows that the peak value of the catalyst, where the XRD pattern reveals that all catalysts are highly crystalline MFI-type framework materials (characteristic peak at $2\vartheta = 7.9°$, 8 ,8°, 23.1°, 23.8°, and 24.3°[1]. However, with increasing alkali concentration during the treatment, the intensity of the sample diffraction peak decreased gradually, indicating that the alkali treatment did not significantly affect the zeolite crystal phase structure. The phase of the ZSM-5 zeolites are maintained after desilication.

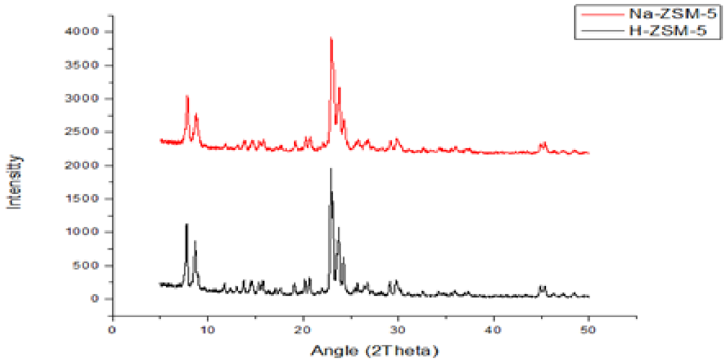

**FIGURE 1** XRD patterns of H-ZSM-5 and Na-ZSM-5

The morphological properties was studied with SEM-EDX. Table 1 shows the results of EDX on H-ZSM-5 and Na-ZSM-5 catalysts. From the results of the analysis, it can be seen that there is a decrease in the quantity of Si content. The H-ZSM-5 catalyst was treated with 1M NaOH so that Na-ZSM-5 was obtained, in which the removal of Si atoms from the zeolite framework.

The results of SEM-EDX analysis showed that the crystallite morphology of H-ZSM-5 did not change much in desilication. This is in accordance with the research conducted previously[3] in Fig. 2 which shows that the crystallite morphology of H-ZSM-5 did not change much in desilication, that there was no significant change even though the crystal size was slightly reduced. The catalyst is in the form of small grains which are Si/Al. It can be seen that the H-ZSM-5 catalyst has micro size pores.

**TABLE 1** EDX Results of H-ZSM-5 and Na-ZSM-5 Catalysts

| | H-ZSM-5 before alkali treatment | | Na-ZSM- after alkali treatment | |
|---|---|---|---|---|
| Element | Wt% | At% | Wt% | At% |
| C | - | - | 28.45 | 38.46 |
| O | 41.31 | 55.24 | 45.92 | 46.61 |
| Na | - | - | 0.64 | 0.45 |
| Al | 2.02 | 1.60 | 1.45 | 0.87 |
| Si | 56.67 | 43.17 | 23.53 | 13.60 |

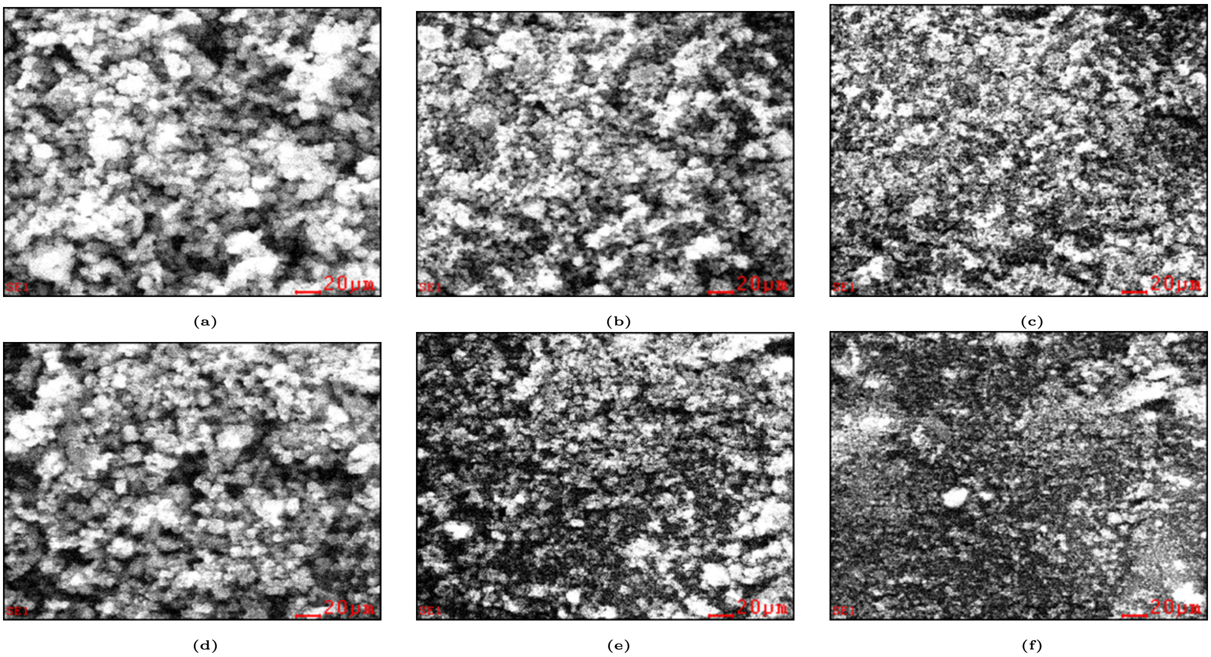

**FIGURE 2** SEM Image of Catalyst: a) H-ZSM-5 5000X, b) H-ZSM-5 10000X, c) H-ZSM-5 20000X, d) Na-ZSM-5 5000X, e) Na-ZSM-5 10000X, f) Na-ZSM-5 20000X

The acidity of the hierarchical ZSM-5 samples was studied by FT-IR pyridine in Fig. 3 and Table 2 shows the acidity calculation. The acidity of the hH-ZSM-5 catalysts could be separated into three peaks at 1442.8, 491.02 and 1545.03 cm-1, which represented weak, medium, and strong acidity, respectively [5] [6] [7] [8] [9]. These three kinds of adsorbed NH3 species could be assigned to NH3 weakly adsorbed on the Lewis acid sites, NH4+ on the Brønsted acid sites, and strong acid absorption on and/or interaction with the dislodged Al, respectively. The amounts of desorbed NH3 for each type of acidic site were calculated and are listed in Table 2 . In this work, the alkali treatment influenced the weak and medium acidity distribution of the ZSM-5 but did not substantially change the strong acidity distribution.

After desilication has been conducted, the number of strong acid sites in the hH-ZSM-5 samples were higher than that of the parent sample has strong total acidity 0.162505 (mmol/g), because some Al species were produced, and the mild acid washing could wash away the Al species located on the external surface of the mesopores. This was attributed to the higher Al content in the alkali-treated zeolites, which gave higher densities of acid sites. The acid site density decreased due to the collapse of the zeolite framework and/or the presence of Al atoms in non-accessible positions or the non-framework octahedral Al of the pore walls.

The textural properties of the hierarchical H-ZSM-5 are shown in Table 3 . The BET surface area (SBET) of hH-ZSM-5 was 357.66 m2/g with 157.48 m2/g of micropore surface area (Smicro). For the hierarchical samples treated

**TABLE 2** Acidity calculation results of catalysts

| Nama sampel | Number of Lewis acid sites (mmol/g) | number of Bronsted acid sites (mmol/g) | Number of acid sites (mmol/g) |
|---|---|---|---|
| H-ZSM-5 | 0.071101 | 0.003642 | 0.074743 |
| Na-HZSM-5 | 0.35124 | 0.003187 | 0.354427 |
| hH-ZSM-5 | 0.161367 | 0.001138 | 0.162505 |

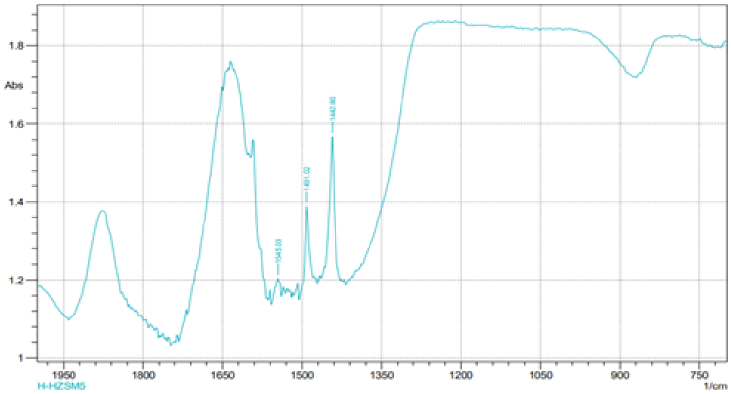

**FIGURE 3** Spektrum FT-IR of hH-ZSM-5 catalyst

with NaOH and HNO3, the mesopore surface area (Smeso) and volume (Vmeso) increased while the Smicro and Vmicro decreased with increasing alkali concentration because of the development of mesopores.

**TABLE 3** Textural properties of hH-ZSM-5

| **Catalyst** | **SBET ($m^2$ $g^{-1}$)** | **Smicro ($m^2$ $g^{-1}$)** | **Smeso ($m^2$ $g^{-1}$)** | **Vmicro ($cm^3$ $g^{-1}$)** | **Vmeso ($cm^3$ $g^{-1}$)** |
|---|---|---|---|---|---|
| H-ZSM-5 | 362.77 | 315.13 | 47.64 | 0.1560 | 0.0890 |
| hH-ZSM-5 | 357.66 | 157.48 | 200.18 | 0.0898 | 0.1412 |

## 4.2 | Results of Plastic Oil Catalytic Cracking With H-ZSM-5 and hHZSM-5

The catalytic cracking of plastic oil was started by preparing PP/LDPE (75:25) % plastic oil variations, namely, 200, 300, 350 and 400 ml and H-ZSM-5 and hH-ZSM-5 catalysts with a mass of 0.5 g. In the catalytic cracking process using FCC (Fluid Catalytic Cracking) reactor, with heating conditions at a temperature of 150-200 oC for 60 minutes.

First, the variation of the oil that will be used for catalytic cracking is prepared, and the variation of the catalyst that will be used. Then put into the FCC reactor, make sure it is tightly closed and sealed to avoid leakage, then heat it for 60 minutes at a temperature of 150-200 $^oC$. The yield result is shown in Figures 4 .

Fig. 4 . shown that the best ratio (b/v) for H-ZSM-5 dan hH-ZSM-5 is on the variant (250 mL/0.5 g) and (350 mL/0.5 g) gives the best result of yield production with 79.6% and 50.8% respectively.

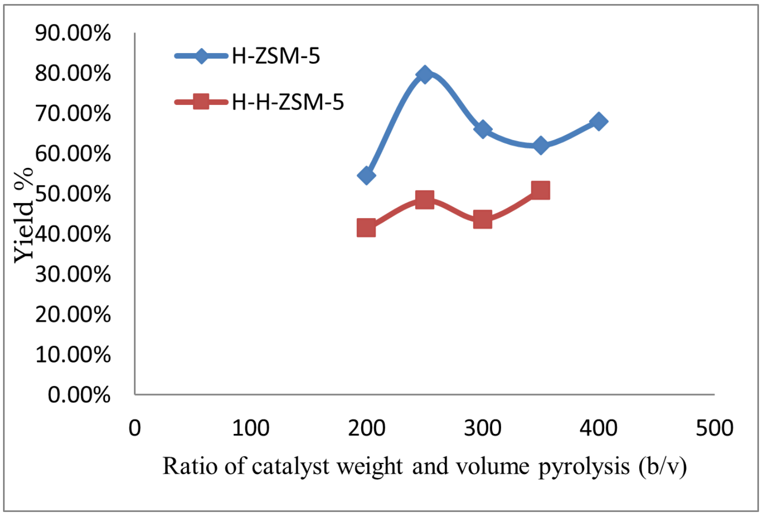

**FIGURE 4** Yield of liquid product on catalytic cracking with H-ZSM-5 and hHZSM-5 catalyst

## 4.3 | Characterization of Catalytic Cracked Oil

Table 4 shows the results of oil analysis carried out with GCMS, cracking using H-ZSM-5 and h-H-ZSM-5 catalysts. While the best variation from H-ZSM-5 is the 2nd variant (250 mL/0.5 g), and from hH-ZSM-5 is the 3rd variant (300 mL/0.5 g), which is shown in the Fig. 5 .

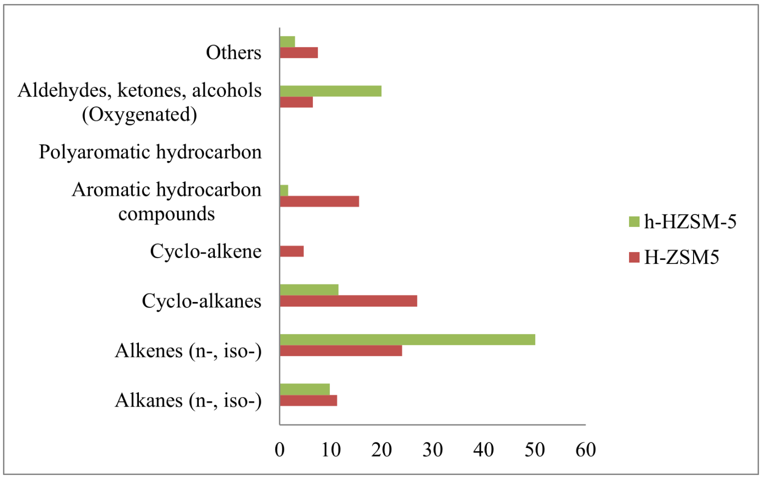

**FIGURE 5** Hydrocarbon Groups Composition after Catalytic Cracking

The length distribution of hydrocarbons in the kerosene/kerosene constituent mixture ranges from a number of carbon atoms C6 to C20, although kerosene usually contains mostly C9 to C16 hydrocarbons. Regardless of the crude oil source or processing history, the main components of kerosene are branched and straight chain alkanes and naphthenes (cycloalkanes), which typically account for at least 70% by volume. Aromatic hydrocarbons in this boiling range, such as alkylbenzenes (single rings) and alkylnaphthalenes (double rings), usually do not exceed 25% by volume of the kerosene flow. Olefins are usually no more than 5% by volume.

The quality of the fuels from cracking with the hH-ZSM-5 catalyst when compared to the H-ZSM-5 catalyst, the use of the hH-ZSM-5 catalyst has high oil quality, with shorter hydrocarbon chains in all variations, namely in the C7 range. -C14 or comparable to kerosene or kerosene. While the H-ZSM-5 has a hydrocarbon range of C6-C22, or still equivalent to diesel or diesel. However, the H-ZSM-5 variation has a higher aromatic compound formation than hH-ZSM-5, so further studies are needed to determine the better oil quality for both types of catalysts.

**TABLE 4** Hydrocarbon Composition Analysis Results of PP/LDPE pyrolysis oil

| No. | PP/LDPE pyrolysis oil (mL) | H-ZSM-5 (g) | hH-ZSM-5 (g) | Hydrocarbon Vomposition Hydrocarbon Range (C5-C14) and/or (C15-C40) |
|---|---|---|---|---|
| 1 | 200 | 0.5 | 0 | C9-C24 |
| 2 | 250 | 0.5 | 0 | C7-C29 |
| 3 | 300 | 0.5 | 0 | C6-C29 |
| 4 | 350 | 0.5 | 0 | C6-C22 |
| 5 | 400 | 0.5 | 0 | C9-C13 |
| 6 | 200 | 0 | 0.5 | C7-C14 |
| 7 | 250 | 0 | 0.5 | C7-C14 |
| 8 | 300 | 0 | 0.5 | C7-C14 |
| 9 | 350 | 0 | 0.5 | C7-C14 |
| 10 | 400 | 0 | 0.5 | C9-C24 |

## 5 | CONCLUSION

In this work, hierarchical ZSM-5 catalysts were prepared by desilication with NaOH The higher acidity of the hierarchical samples influenced the conversion of plastic fuels turned into kerosene and diesel. The suitable mesopores introduced by alkali treatment increased the yield of aromatics due to the enhanced mass transfer of reactants. Finally, the H-ZSM-5 gave higher yields of aromatics than hierarchical H-ZSM-5 (hH-ZSM-5), but the hierarchical produce the hydrocarbon range shorter and have higher aromatic compound.

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## CREDIT

Conceptualization, Methodology, Writing - original draft preparation, and Supervision: Lenny Marlinda; Formal analysis and investigation: Silvia Dwi Cahyania, Bagas Rimawan, Yuli Evrianti; Writing - review and editing: Rahmi, Lenny Marlinda; Funding acquisition: Lenny Marlinda; Resources: Sutrisno, Heriyanti, Lenny Marlinda.

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