



Research article

Conversion of polypropylene-derived crude pyrolytic oils using hydrothermal autoclave reactor and Ni/aceh natural zeolite as catalysts



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ABSTRACT

The accumulation of plastic waste has urged researchers to develop methods of waste conversion into valuable products, which is fuel. This study aimed to synthesize Ni embedded onto Aceh natural zeolite (Ni/Aceh-zeolite) as a cheap catalyst which could be used in the reforming process to improve the quality of oil produced from polypropylene (PP) pyrolysis. Ni/Aceh-zeolite was synthesized from $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and acid-activated natural zeolite through impregnation and calcination. The catalyst was found to have particle sizes ranging from 100 to 200 nm of 20 wt% Ni content. The reforming process using Ni/Aceh natural zeolite with Ni loading of 15 wt% yielded the highest amounts of liquid product (yield = 65%) and gasoline fractions ($\text{C}_5\text{--}\text{C}_{12}$, 96.71%). However, the highest high heating value of 45.467 MJ/kg was found in the liquid product obtained with 20% Ni/Aceh-zeolite. In conclusion, Ni/Aceh-zeolite could be used in the reforming process of PP pyrolysis-derived oil, which could reach a quality similar to that of commercial gasoline.

1. Introduction

Plastic production has skyrocketed since its initial commercialization, reaching up to three million tons of global production [1–5]. Consequently, it threatens the environment due to the complicated decomposition. Even though plastic recycling is thought to be the solution, the plastic waste is required to be in a specific form (such as seeds/pellets, granules, powders, or fractions), hindering its large-scale implementation [6–8]. Therefore, researchers have proposed effectively converting plastic waste into fuels as an alternative solution [9–12]. Pyrolysis, the thermochemical conversion that involves the catalyst in the degradation process, is a common technique for converting plastic waste into energy in the form of solid, liquid, and gaseous fuels [13–16]. Pyrolysis allows the breakdown of long-chain hydrocarbon plastic into small molecules of liquid and gaseous hydrocarbons. In previous research, plastic waste-derived

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oil had similar properties to commercial diesel [17]. However, direct catalytic cracking of waste plastics has many drawbacks that have hindered its commercial success. The first is the difficulty of recovering the catalyst after use, which increases operating costs. Moreover, direct contact with plastic waste leads to rapid deactivation of the catalyst due to the deposition of carbonaceous material and poisoning effects from foreign substances and impurities such as chlorine, sulfur, and nitrogen species that may be present in the plastic waste [18]. Separation of the catalytic reforming reaction from the pyrolysis stage can therefore be used to overcome these problems.

A catalytic reforming process is required afterwards to obtain a fuel with high octane values [19–21]. The common catalysts include platinum (Pt), palladium (Pd), copper (Cu), and cerium (Ce), which have been previously supported on γ - Al_2O_3 [22–25]. However, both of the foregoing metals are expensive. In the reforming process, the type of catalysts and reaction pathways determine the quality of the reformed oil. Zeolite catalysts, in combination with metals, have been typically used in the reforming processes [26–30]. Mo/zeolite, commercial Y-zeolite, and natural zeolite catalysts were used to convert HDPE plastic into hydrocarbon. All catalysts were able to convert the plastic feed into gaseous and liquid hydrocarbons, showing low coking levels, while the lowest coke was produced over the Co/Z catalyst [31]. Alternatively, nickel has been known as a transition metal with less price than Pd and Pt. The metal could be embedded into supporting materials to enhance its catalytic performance, which is zeolite comprising Al_2O_3 and SiO_4 [32–34].

Previous research has suggested the high selectivity of Ni/Aceh natural zeolite catalyst [35] in the cracking reaction and its ability to prevent coke formation on the catalyst surface. Natural zeolites are known for their acidic properties, making them efficient in acid catalysis. It has also attracted attention as an effective catalyst for the conversion of plastic waste and high yields of hydrocarbons. Nickel-impregnated Aceh natural zeolites (Ni/Aceh-zeolite) are cheap and have been used in hydrogen production with satisfactory results, so they were nickel-filled in this study. Furthermore, nickel performed well in a microwave-catalytic water glycerol solution process using supported nickel to produce [36,37]. According to this study, the Ni/Aceh-zeolite provided synergistic improvements in oil yield in hydrocarbon production. However, the use of catalysts is the main cost burden for recycling plastic waste. Reducing the catalyst cost for small-scale applications in developing countries like Indonesia is an exciting challenge. Aceh natural zeolites, which can be found in many places, including Aceh Province, Indonesia, might be used as a candidate for this purpose instead of commercial catalysts. A large number of papers have been published describing the catalytic pyrolysis of plastics [38–41]. There are only a few papers utilizing Aceh natural zeolite as a catalyst feedstock to convert plastic wastes. Therefore, in this study, the Ni/Aceh-zeolite was used in the PP reforming catalyst pyrolysis products to measure the catalytic effect.

In addition to addressing these topics, we investigate the catalytic activity of the nickel-impregnated Aceh natural zeolite catalyst in the reforming process to improve the quantity and quality of the pyrolysis oil products. To assess the product's quality, the oil's characteristics, such as viscosity, density, and calorific value, acquired by catalytic reforming, are thoroughly studied. The impregnation loading of nickel in the catalytic hydrothermal autoclave reactor was varied for this investigation. X-ray diffraction (XRD), scanning electron microscopy-energy dispersive X-ray (SEM-EDS), and transmission electron microscopy (TEM) were used to examine the catalytic properties. In addition, Fourier transform infrared spectroscopy (FT-IR), and gas chromatography-mass spectrometry (GC-MS) were used to examine the product's functional groups and chemical composition.

2. Materials and methods

2.1. Materials

Aceh natural zeolite was sourced from Aceh Besar, Indonesia and PP was obtained from the landfill in Java village, Banda Aceh, Indonesia, which was activated through acids before being used. HCl 3 M solution (37%, Merck), $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ powder (99.99%, Merck), and N_2 gas (99%, PT. Aneka Gas Industri). All chemicals used herein were purchased in analytical grade and used without further treatment.

2.2. Synthesis of Ni/aceh natural zeolite catalyst

The natural zeolite was ground and sieved (250 mesh) to achieve a uniform size. The zeolite was then washed with demineralized water and oven-dried at 120 °C for 12 h. Subsequently, it was acid-activated in a 3 M HCl solution for 6 h. The acid-activated zeolite was then filtered and rinsed with demineralized water repeatedly to remove the Cl^- ion. The residue was dried at 120 °C and sieved to achieve a consistent size. Then, the samples were labeled as acid-activated natural zeolite (ZAA).

The preparation of Ni/Aceh natural zeolite catalyst followed the method from a previously published report [42]. Briefly, Ni ($\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was firstly dissolved in 10 mL of water and subsequently added with 10 g ZAA for the impregnation. The mixture was stirred for 6 h at room temperature. The mixtures were then oven-dried at 115 °C for 12 h. The resulting powder was then thoroughly ground and calcined at 350 °C in the air for 2 h to form Ni/Aceh natural zeolite. The weights of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were varied as 0.5 g, 1.5 g, and 2 g to yield 5% Ni/Aceh natural zeolite, 15% Ni/Aceh natural zeolite, and 20% Ni/Aceh natural zeolite, respectively.

2.3. Characterization

TEM was employed to investigate Ni/Aceh natural zeolite nanoparticles using a Philips/FEI Tecnai 20G2 S-Twin TEM apparatus (Kyoto, Japan). The catalyst was mixed with a volatile liquid before being dripped onto the grid and subsequently scanned with a high-energy electron beam (at 120 kV). Hitachi TM300 (Krefeld, Germany) was used for SEM and EDX. The Shimadzu XRD-7000 diffractometer (Kyoto, Japan) was used to determine the crystalline characteristics of the Ni/Aceh natural zeolite-prepared catalysts.

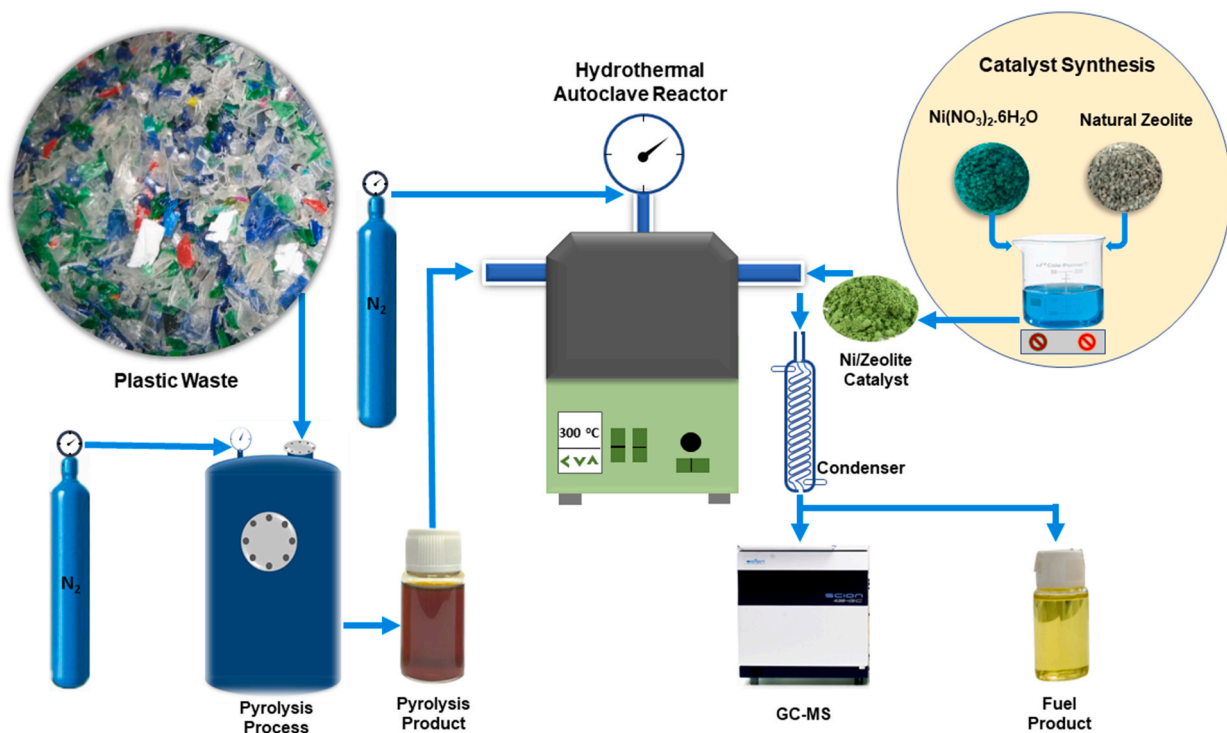


Fig. 1. Experimental workflow of reformed oil production from PP waste through pyrolysis.

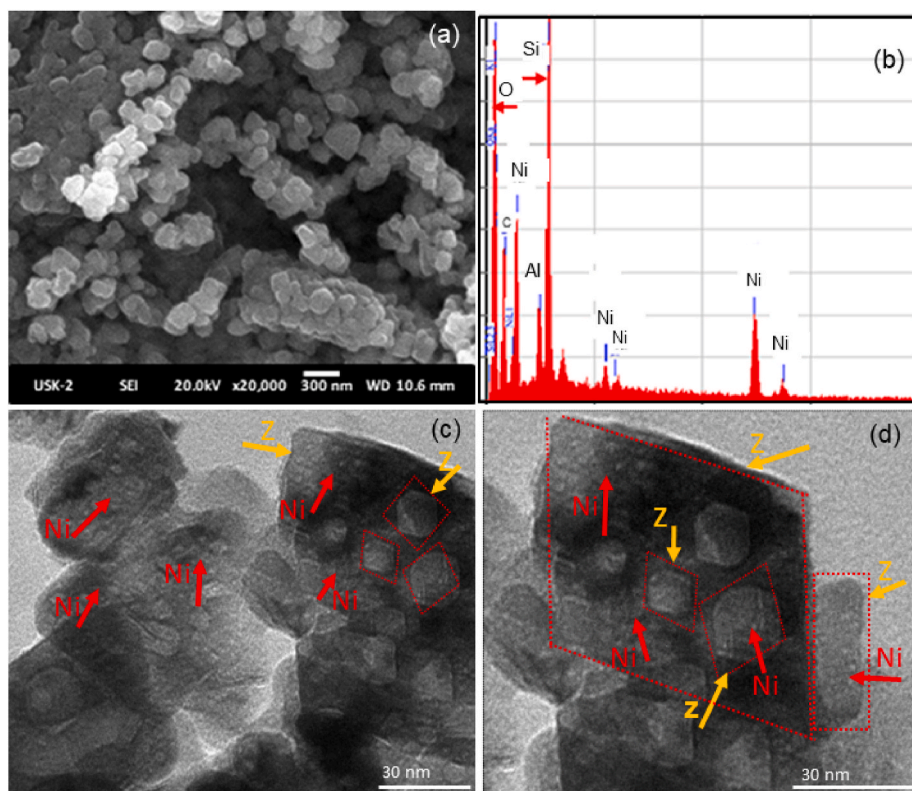


Fig. 2. (A) SEM images of Ni/Aceh natural zeolite (Ni/ZAA:20 wt% Ni); (b) EDX spectrum Ni/ZAA (c) TEM images of Ni/ZAA; (d) TEM images (arrow: view of Ni on zeolite surface, Z: view of some zeolite shape), 20 wt% Ni.

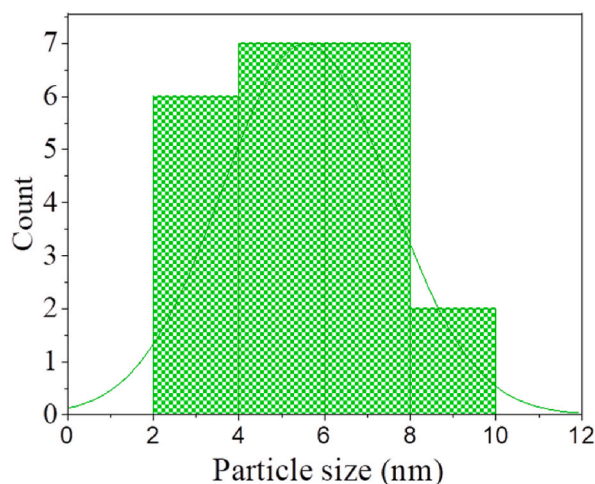


Fig. 3. Average particle diameters of Ni/Aceh natural zeolite catalysts at a 20 wt% Ni prepared at a calcination temperature of 350 °C (based on TEM images from Fig. 2d).

2.4. Pyrolysis process and reforming reaction

A plastic chopping machine was used to reduce the size of plastic waste before being inserted into a pyrolysis reactor concomitant to the ambient air evacuation by continuous N₂ flow. The process was carried out at 450 °C for 3 h. The pyrolysis output was allowed to cool down until the gas products were removed. The liquid product was then fed into a batch reactor (hydrothermal autoclave reactor) with a Ni/Aceh natural zeolite catalyst for the reforming process. A catalyst was introduced into the reactor with 10 wt% oils from pyrolysis. Thereafter, the N₂ gas was injected into the batch reactor to purge the air. The reforming process was run at 280 °C, where the resulting product was flown through a condenser. The effect of Ni loading was investigated by using Ni/Aceh natural zeolite catalyst with Ni loading variation (5, 15, and 20% Ni/Aceh natural zeolite) in the reforming process under the same aforementioned conditions.

The GC–MS analysis using Shimadzu QP2010 PLU (Kyoto, Japan) was employed to determine the composition of the liquid product. A Cannon Fenske viscometer was used to measure the viscosity of pyrolysis and reforming products. The sample was collected using an adaptation of the ASTM D445 standard procedure. The pyrolysis product density was determined using a pycnometer and the ASTM D1298 standard. The combustion of pyrolysis oil from PP was tested using a bomb calorimeter (Automatic calorimeter K88890) following ASTM D240. The test involved burning 5 mL of the sample in a platinum crucible and an “iron coil” under controlled conditions. The heat of combustion was calculated from the beginning of the pre-combustion observation throughout the combustion process and afterwards, with appropriate allowances for thermochemical correction and heat transfer. The results of the generated product were compared with the properties of commercial diesel or gasoline. The experimental workflow for the pyrolysis of PP plastic waste followed by the reformation of the crude oil using Ni/Aceh natural zeolite catalyst has been presented in Fig. 1.

3. Results and discussion

3.1. Characteristics of the Ni/aceh natural zeolite

3.1.1. SEM–EDX and TEM images

SEM–EDX was employed to investigate the surface morphological structure, composition, and particle sizes of the catalyst, which the results have been presented in Fig. 2.

The catalyst appeared to have a hexagonal particle morphology that is mostly irregular in sizes ranging from 100 to 200 nm, shown in Fig. 2a. According to the EDX analysis in Fig. 2b. The catalyst contained 28.55 mass % Ni (13.21 atomic % Ni). The Ni elements were anchored on the Ni/ZAA surface. Another component is composed of Al, Si, Na, Mg, and K. These findings are in accordance with the fact that zeolite is the source of Al, Si, and K, hence confirming the successful Ni impregnation onto zeolite. TEM images indicate the uniform distribution of Ni metal on the zeolite surface (Fig. 2 (c, d)). The uniform distribution is attributed to the highly dense active sites of zeolite, as suggested by a study investigating Ni/ZAA [43].

As an active site, nickel was loaded on the zeolite powder to promote gasoline production. Fig. 3 shows the particle size distribution of the nickel-loaded ZAA nanoparticles. TEM observation revealed that Ni metal with an average particle size of about 2–8 nm was anchored after calcined in the air under 350 °C. During the oxidation step in the air under mild conditions, one could create a thin layer of NiO on the surface of ZAA.

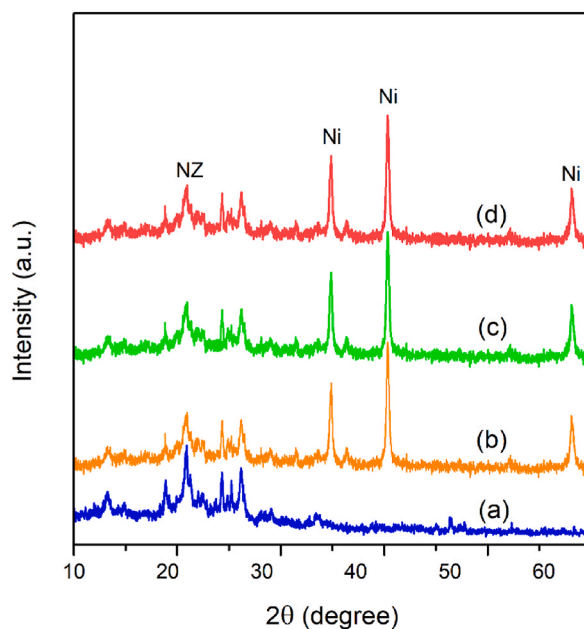


Fig. 4. XRD patterns of (a) Aceh natural zeolite (ZAA); (b) 5% Ni/ZAA; (c) 15% Ni/ZAA, and (d) 20% Ni/ZAA.

Table 1
BET catalyst surface area analysis.

Ni on ZAA	Surface area (m ² /g)	Pore volume (cm ³ /g)	Average Pore Diameter (nm)
ZAA	101	0.21	14.1
Ni (5%)	99	0.31	12.6
Ni (15%)	88	0.34	10.9
Ni (20%)	53	0.37	9.7



Fig. 5. Liquid products from (a) PP pyrolysis and reforming process using Ni/ZAA with Ni loadings of 5.0 wt% (b), 15% wt.% (c), and 20 wt% (d).

3.1.2. XRD patterns

The XRD patterns of Aceh natural zeolite and Ni/Aceh natural zeolite with various Ni loadings have been presented in Fig. 4(a–d). The XRD diffractogram of each sample was compared with the standard compound of Ni and natural zeolite. Additional crystalline peaks emerged following the Ni embedment. The Ni compounds have three primary peak intensities at $2\theta = 43.3^\circ$, 37.25° , and 62.9° (JCPDS: 00-047-104). Taken altogether, the XRD patterns corroborate the previous findings of the successful Ni embedment.

According to the Scherrer equation, the nickel particle size of 5% wt. Ni was 5.4 nm and increased slightly to 6.5 nm and 8.2 nm for the Ni containing 15% and 20% wt. This may be due to decreased inter-particle pores, suggesting the nickel was less dispersed on the surface [44].

3.1.3. Brunauer-emmett-teller (BET) of the prepared ZAA and Ni/ZAA

The BET surface area, pore volume, and average pore diameter of ZAA before and after Ni impregnation are displayed in Table 1. As

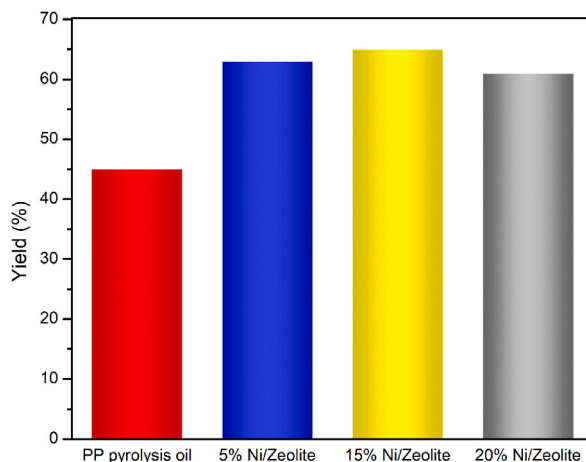


Fig. 6. Yield of oils produced from PP pyrolysis with and without reforming process using Ni/ZAA.

Table 2

Properties of oils produced.

Sample Specification	PP pyrolysis oil	5% Ni/ZAA	15% Ni/ZAA	20% Ni/ZAA
High heating value (MJ/kg)	43.214	43.320	44.173	45.467
Density (kg/m^3)	880	768	728	736
Viscosity (cSt)	0.855	0.804	0.755	0.772

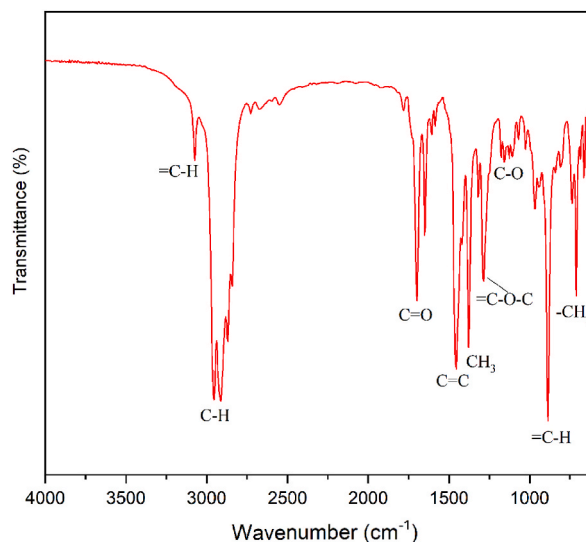


Fig. 7. FTIR spectrum of the reformed oil deriving from PP waste.

a direct result of the impregnation process, the surface area of the catalyst was reduced because impregnated nickel acted as a block. However, the addition of Ni resulted in an increase in pore volume, which indicated that the material's catalytic efficiency had been enhanced.

3.2. Catalytic performance of Ni/aceh natural zeolite and liquid characteristics

3.2.1. Color appearances of the liquid products

Color appearances of the liquid product obtained from the PP pyrolysis and that with the reforming process using Ni/Aceh natural zeolite have been presented in Fig. 5(a–d). The liquid product from PP pyrolysis appeared in a brownish color. The appearance transformed into a lighter tone color following the reforming process with Ni/Aceh natural zeolite catalyst, especially with that higher

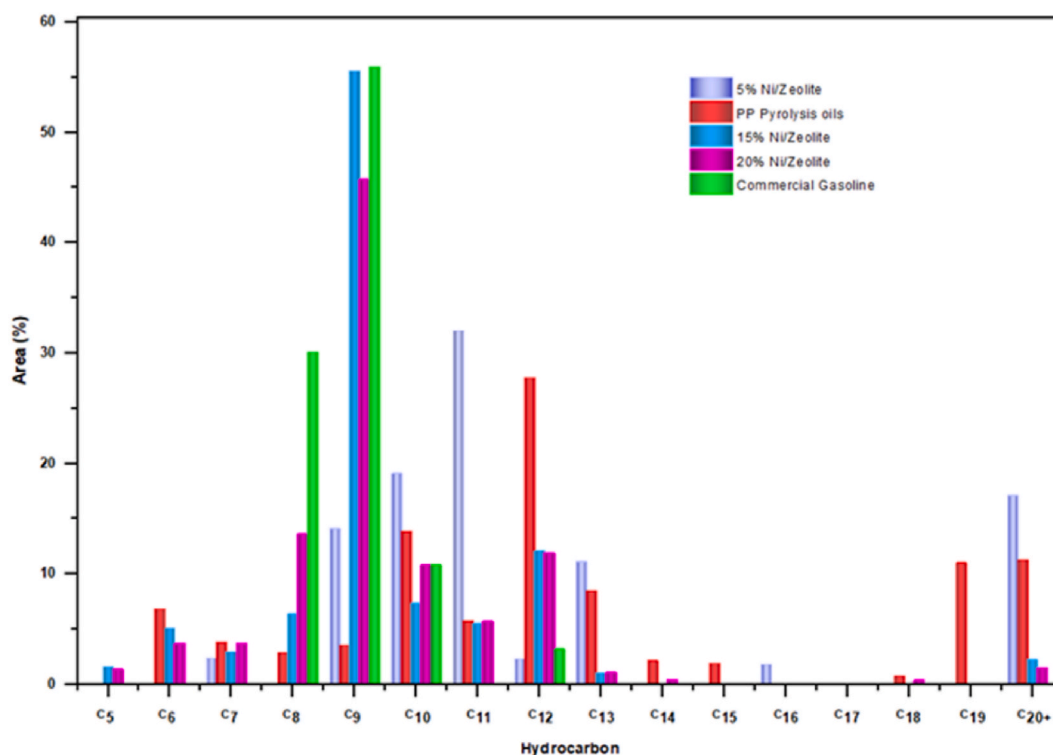


Fig. 8. Hydrocarbon numbers of pyrolysis and reforming oils (PP pyrolysis oils, 5% Ni/Aceh natural zeolite, 15% Ni/Aceh natural zeolite, 20% Ni/Aceh natural zeolite) and commercial gasoline.

Ni loadings. As suggested by a previous study [45], PP-derived oil with light color has predominantly consisted of molecules with short carbon chains.

3.2.2. Oil properties

As can be seen from Fig. 6, the yield of oils produced from PP pyrolysis without the reforming process was obtained at 45%. The yield of oil products increases with increasing Ni loading. This increase is due to the increase in active sites as the catalyst loading increases. In terms of hydrocarbons, it progressively increased from 62.93% (5 wt%) to 65.1% (15 wt%) and decreased to 60.83% at 20 wt% Ni loading [30,46]. The best results were obtained with a catalyst loading of 15% by weight. The increased catalyst loading to 20% by weight slightly decreased the deoxygenation performance. This shows that excessive Ni load can cause side reactions such as cracking and polymerization [47]. This can be seen from the slight increase in gasoline and heavy hydrocarbons by 20% by weight. Therefore, a catalyst loading of 15% by weight is the most suitable because it produces optimal plastic oil conversion (65.1%).

Yield, high heating value (HHV), density, and viscosity of the resultant oils have been presented in Fig. 6 and Table 2. Higher Ni loading resulted in higher liquid product yield, except for 20% Ni loading. Higher Ni loading might result in a higher gas product which consequently leads to a lower liquid product. HHV represents the enthalpy change or heat released resulting from combustion. The HHV of oil produced from PP pyrolysis was 43.214 MJ/kg. The addition of Ni/Aceh natural zeolite increased the HHV to 43.320–45.467 MJ/kg. Liquid products obtained herein had similar HHV values with that of commercial diesel and gasoline fuels [48, 49].

3.2.3. FTIR profile the reformed oil

The FTIR spectrum of the liquid produced from the pyrolysis of PP, followed by the reforming process using Ni/Aceh natural zeolite (Ni loading 15%), has been presented in Fig. 7. A spectral peak at 3074.53 and 887 cm^{-1} are assigned to the stretching and bending vibration of =C–H. Spectral bands at 2951.09 and 2916.37 cm^{-1} are assigned to the stretching vibration of C–H. The presence of C=O group was observable by the stretching vibration appearing at 1697.36 and 1647.21 cm^{-1} . A typical spectral band of aromatic C=C was observed at 1377.17 and 717.52 cm^{-1} . The presence of methyl –CH₃ and methylene –CH₂– was indicated by spectral peaks at 1377.17 and 717.52 cm^{-1} , respectively. Collectively, the FTIR profile suggests that the main products of the PP pyrolysis, followed by the reforming process using Ni/Aceh natural zeolite catalyst, were alkanes, alkenes, and aromatics, according to the FTIR spectra.

The combination of acidic sites on zeolite and the selectivity of Ni is responsible for the improved cracking process of long-chain hydrocarbons into lighter molecules [50,51]. The reforming process involved the cracking of PP-derived long-chain hydrocarbons and was subsequently followed by hydrocarbon isomerization reaction, H and C atoms rearrangements, and cyclization. In addition, the formation of alkanes, alkenes, and aromatic compounds might occur concomitant to intramolecular attacks on the double bond.

3.2.4. GC–MS profile of the reformed oil

The highest peak area (7.26%) observed in the chromatogram of pyrolysis liquid product belonged to C₉H₁₈. Following the reforming process with Ni/Aceh natural zeolite loaded with 5%, 15%, and 20% Ni, the oils became predominated by C₁₀H₂₀ (9.55%), C₉H₁₈ (25.14%), and C₉H₁₈ (24.54%). Comparatively, commercial gasoline had C₈H₁₀ hydrocarbons with a peak area of 20.8%. All of the aforementioned oils had gasoline-grade of hydrocarbon components (C₅–C₁₂).

Hydrocarbon numbers of the liquid products, along with that of commercial gasoline, have been presented in Fig. 8. The reformed product using a 15% Ni/Aceh natural zeolite catalyst had a total area of C₉ that was similar to that of commercial gasoline (55.56% and 55.89%, respectively). Based on the accumulative number of C₅–C₁₂ fractions, the highest was found in the reformed oil obtained with 15% Ni/Aceh natural zeolite (96.71%), followed by that obtained with 20% Ni/Aceh natural zeolite (96.64%), and 5% Ni/Aceh natural zeolite (69.95%). Meanwhile, crude oil produced from PP pyrolysis only had the accumulative number of C₅–C₁₂ fractions of 64.42%. As for commercial gasoline, the accumulative number of C₅–C₁₂ fractions reached 96.81%.

4. Conclusion

To improve the production of gasoline fractions in liquids obtained from PP pyrolysis, Ni/Aceh natural zeolite catalysts with different Ni loadings (5%, 15%, and 20%) were used in the reforming process. The catalysts exhibited a high crystallinity structure with nanoscale dimensions of 30–200 nm. EDX and XRD analyses confirmed the successful embedment of Ni into the acid-activated zeolite. Gasoline fractions (C₅–C₁₂) improved significantly and became similar to that of commercial gasoline following the reforming process with Ni/Aceh natural zeolite catalyst. Reformed oil with the highest gasoline and yield percentage was obtained from a process employing 15% Ni/Aceh natural zeolite. Nonetheless, the HHV of reformed oil obtained with 15% Ni/Aceh natural zeolite was lower than that obtained with 20% Ni/Aceh natural zeolite.

Author contribution statement

Husin Husin: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Mahidin, Marwan: Conceived and designed the experiments; Analyzed and interpreted the data.

Fahrizal Nasution: Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Erdiwansyah, Ahmadi, Syawaliah Muchtar, Rizalman Mamat: Analyzed and interpreted the data; Wrote the paper.

Firda Tirta Yani: Performed the experiments; Analyzed and interpreted the data.

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Data availability statement

Data include in article/supp. Material/referenced in article.

Additional information

No additional information is available for this paper.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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