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Short communication

Catalytic conversion of furfural extract from lubricating oil extraction unit over the shaped and promoted HY catalysts to valuable petroleum products

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ABSTRACT

Catalytic conversion of a furfural extract from the extraction unit of lubricating oil to valuable products was investigated over the shaped HY extrudates using bentonite as binder (HY/bentonite) and zinc and nickel promoted HY/bentonite catalyst (Zn-Ni-HY/bentonite) in a bench scale fixed bed reactor. The catalysts were characterized by XRF, XRD, SEM and nitrogen adsorption-desorption. At 550 °C and LHSV of 9.2 h⁻¹, 7.3 wt% gasoline was yielded over HY/bentonite catalyst, which was significantly increased (ca. 42%) over Zn-Ni-HY/bentonite catalyst. Besides, notably enhanced yield of light and heavy naphtha, kerosene, gas oil and lubricating oil can be distilled from the overall liquid products over Zn-Ni-HY/bentonite catalyst (90.5 wt%) than over HY/bentonite catalyst (77.6 wt%). The obtained petroleum products with the satisfied characteristics can be blended after hydrodesulfurization processing.

1. Introduction

Furfural extract is a by-product from the extraction unit of lubricating oil by furfural to improve the viscosity index [1,2]. The extract, characterized by a black color and high viscosity, contains lubricating oil residues and is rich in highly condensed polycyclic aromatics. The use of this extract is very limited, though some applications have been mentioned (e.g., in the tire industry, as asphalt blends, and for rubber and plastic manufacture) [1,3]. Thus, it is of high interest to find alternative high value outlets, for instance, to produce high-quality petroleum products. Thermal cracking of heavy petroleum fractions is widely applied in the petrochemical industry. In this process, larger hydrocarbons are cracked to smaller molecules followed by distillation to obtain light petroleum products [4,5]. Generally, cracking of the aromatic components has lower conversion and the gasoline yield is lower than the yields of paraffins, naphthenes and olefins [6]. As such, a high cracking temperature is required. In contrast, catalytic cracking allows control over the cracking reactions and typically leads to higher gasoline yields [7]. In addition, the gasoline fraction typically also has a higher octane number than those obtained from thermal cracking [8,9]. Zeolite based catalysts are often used in the catalytic cracking process due to their distinct crystalline structure,

large internal surface areas, uniform pores with multiple sizes, strong acidic sites, high thermal stability, and the ability to absorb the hydrocarbons [10]. Particularly Y zeolites (e.g., NaY) are the preferred catalysts for catalytic cracking and are often transformed into HY zeolite by ion exchange [11].

This submission presents a demonstration study on catalytic conversion of a furfural extract over the shaped and promoted HY Zeolite based catalysts to produce useful petroleum products. The catalysts were synthesized and characterized in detail and subsequently tested in a fixed bed configuration. To the best of our knowledge, this is the first study of the utilization of a furfural extract via catalytic conversion approach reported in the literature.

2. Experimental

2.1. Catalyst preparation

NaY zeolite synthesis: Fused kaolin was prepared by mixing kaolinite (Table S1) and sodium hydroxide solution followed by calcination at 850 °C for 3 h. The resulting fused kaolin was then added to sodium silicate solution at a pH value of 13.3 under stirring for 1 h at 50 °C. The mixture was aged at 50 °C for 24 h and at 100 °C for 48 h, followed by

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filtration and washing. The filter cake was finally calcined at 500 °C for 1 h to obtain NaY zeolite.

HY zeolite synthesis: The as-prepared NaY zeolite was mixed with an ammonium chloride solution under stirring to form NH₄Y zeolite. The mixture was filtered and the filter cake was dried at 100 °C for 6 h followed by the treatment with oxalic acid at room temperature for 8 h [12]. After aging, filtration and washing, the filter cake was dried at 100 °C for 6 h followed by calcination at 550 °C for 5 h to obtain HY zeolite.

The shaped HY catalyst preparation: Generally, zeolite powder is shaped into pellets in industry by using natural clays (e.g., bentonite) as binders [13]. As such, the as-prepared HY zeolite was mixed with bentonite (Table S1) and deionized water to obtain a homogenous paste. Cylindrical shaped samples with a diameter of 2 mm were obtained by processing the paste in a mechanical extruder, followed by drying at 100 °C for 24 h and cutting into particles with 4–8 mm length. The shaped HY catalyst using bentonite as the binder (termed as HY/bentonite) was obtained by calcining the above extrudates at 600 °C for 2 h.

The promoted HY catalyst preparation: Zinc and nickel promoted HY/bentonite catalyst (termed as Zn-Ni-HY/bentonite) was prepared by adding HY to an aqueous solution of Zn(NO₃)₂ and Ni(NO₃)₂ at 40 °C under stirring for 2 h. The slurry was filtered and the filter cake was dried at 110 °C for 2 h followed by calcination at 600 °C for 3 h. The loading of Zn and Ni in the Zn-Ni-HY/bentonite catalyst was 2.6 wt% and 0.6 wt%, respectively.

The detailed procedure and parameters can be found in the SI.

2.2. Catalyst characterization

The sodium content of the catalysts was determined according to ASTM D-1428-64 by using a flame photometer. X-ray fluorescence (XRF, Spectro XEPOS 2010) was used to determine the silica and alumina content in the catalysts. X-ray diffraction measurements were carried out on a Shimadzu SRD 6000. The bulk crushing strength of the catalysts was determined according to an ASTM method (ASTM D-7084) on a CRUSH BK. The morphology of the catalysts was determined by scanning electron microscope (SEM, Tescan Orsay Holdin). Nitrogen physisorption was performed to determine the surface area and pore volume of the catalysts. The bulk density was determined according to ASTM D-4164 using a Quantachrom Autotap.

2.3. Catalytic conversion of furfural extract

The fluid catalytic cracking (FCC) is the most important chemical conversion process in industry which can be adopted for catalytic conversion of furfural extract. In this demonstration, catalytic conversion of furfural extract (Table 1, the residue obtained from the extraction unit of a medium lubricating oil in the Al-Dora refinery) was performed on a dedicated bench scale unit (Fig. S1, details are in SI), aiming for a rapid catalyst screening and the production of the liquid products for the detailed analysis and distillation. The reactions were carried out at a range of temperatures (T, 450–600 °C) and liquid hourly space velocities (LHSV, 5.12–17.4 h⁻¹). A time on stream (TOS) of 30 min was used, due to the fact that this is the minimum reaction time to obtain a reasonable mass balance (> 90%) and enough amount liquid products. The products were condensed at –20 °C and separated

Table 1

Characteristics of the furfural extract obtained from the extraction unit of a medium lubricating oil in the Al-Dora refinery.

Density at 37 °C (g cm ⁻³)	Pour point (°C)	API gravity	Kinematic viscosity		Conradson carbon residue (%)	Sulfur (wt%)	Initial boiling point (°C)
			At 40 °C (c.st.)	At 80 °C (c.st.)			
1.05	12.9	10.4	410	36	3.45	4.5	410

in the gas-liquid separator. The gaseous products were analyzed by gas chromatography (Pye Unicam, England). The gas yield was calculated using Eq. (1).

$$\text{Gas yield} = \frac{\text{Weight of gas products}}{\text{Total weight of furfural extract fed}} \times 100\% \quad (1)$$

The liquid products were transferred to a distillation unit (details can be seen in Section 2.4) to obtain gasoline fraction (T < 175 °C). The gasoline yield was calculated using Eq. (2).

$$\text{Gasoline yield} = \frac{\text{Weight of gasoline products}}{\text{Total weight of furfural extract fed}} \times 100\% \quad (2)$$

After an experiment, the used catalysts were taken from the reactor and weighed before burning off the coke at 680 °C for 1 h. The coke deposition was calculated by the difference. The coke yield was calculated using Eq. (3).

$$\text{Coke yield} = \frac{\text{Weight of coke deposition}}{\text{Total weight of furfural extract fed}} \times 100\% \quad (3)$$

Due to the molecular complexity of the furfural extract, it is not possible to obtain detailed information on the exact molecular composition. Accordingly, only the conversion of the furfural extract to gasoline products was considered to calculate the conversion of furfural extract using Eq. (4), assuming that the converted furfural extract was transformed into the gasoline products, permanent gases and coke deposited on the catalyst.

$$\text{Conversion} = \frac{\text{Sum of the weight of gasoline, gas and coke}}{\text{Total weight of furfural extract fed}} \times 100\% \quad (4)$$

2.4. Distillation and the analysis of the distilled fractions

The overall liquid products obtained from catalytic conversion of the furfural extract over HY/bentonite and Zn-Ni-HY/bentonite catalysts at 550 °C and an LHSV of 9.2 h⁻¹ were fractionated in the laboratory distillation unit (Micro TOP-P device) according to ASTM 5236 to produce light naphtha (up to 75 °C), heavy naphtha (75–175 °C), kerosene (175–240 °C), gas oil (240–320 °C), and lubricating oil (320–425 °C). The details and operation of the distillation unit are described in the SI.

The sulfur content was determined according to ASTM D-7039 using a Sulfur Analyzer (Sindie OTG, USA). The specific gravity was determined according to ASTM D-4052 by using a DMA4500 density meter (Anton Paar, USA). Determination of the heat of combustion was done according to ASTM D-240 using a Parr 1266 Isoperibol Bomb Calorimeter (USA). The viscosity was determined using a TVB 445 viscometer (Tamson, France). The PONA test was done according to ASTM standard D-6729 using an Agilent 7890 AGC (USA).

3. Results and discussion

3.1. Characterization of the catalysts

The surface chemistry of the prepared NaY zeolite was measured by FT-IR (Fig. 1) and is consistent with that for a reference NaY zeolite [14]. The intensive bands at 443.6–451 cm⁻¹ (assigned to the bending vibrations of the internal tetrahedral TO₄ (T = Al or Si)), 542–569 cm⁻¹

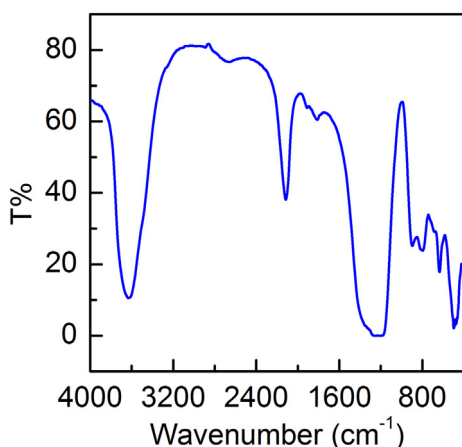


Fig. 1. FT-IR spectrum of NaY Zeolite.

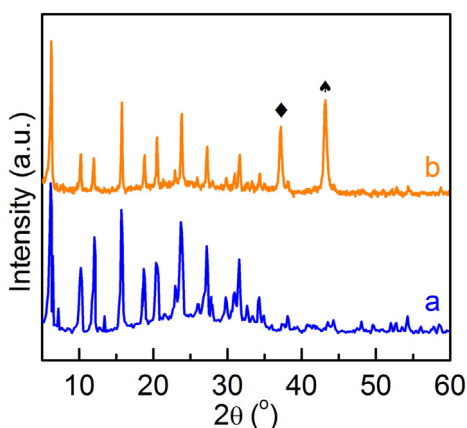


Fig. 2. XRD spectra of (a) NaY Zeolite and (b) Zn-Ni-HY/bentonite catalyst.

(assigned to double ring external linkage), $677\text{--}759\text{ cm}^{-1}$ (assigned to symmetrical stretching), $972.1\text{--}1026.1\text{ cm}^{-1}$ (assigned to asymmetric stretch), 1637.6 cm^{-1} (assigned to covalent bond vibration) and $3442.9\text{--}3473.8\text{ cm}^{-1}$ (assigned to the bridging OH group of Brönsted acidity (Si-O(H)-Al) [15,16] can be observed. The crystal structure of the prepared NaY zeolite was further checked by X-ray diffraction (Fig. 2) and is in accordance with that for a NaY zeolite [17]. The relative crystallinity of NaY zeolite was determined by comparing the XRD data given above with a reference [18, 19] and was 88.7%. Furthermore, the bulk density of the prepared NaY zeolite was 0.576 g cm^{-3} , which is in the range of $0.5\text{--}1\text{ g cm}^{-3}$ as reported by Hansford et al. [20]. The total pore volume of the NaY zeolite catalyst was $0.254\text{ cm}^3\text{ g}^{-1}$ and the specific surface area was $454\text{ m}^2\text{ g}^{-1}$. The latter is also in the range of $300\text{--}700\text{ m}^2\text{ g}^{-1}$ reported for NaY zeolites [21]. Thus, all of the above characteristics indicated that the synthesis of NaY zeolite was successful.

The ion exchange of NaY zeolite with ammonium chloride significantly reduced the Na content of the prepared zeolite from 13.7 wt% (based on Na_2O) for the NaY zeolite to 2.4 wt% (based on Na_2O) on the HY zeolite (Table S2). The specific surface area and pore volume of the HY zeolite are $290\text{ m}^2\text{ g}^{-1}$ and $0.127\text{ cm}^3\text{ g}^{-1}$, respectively, which are lower than for the parent NaY zeolite and is likely due to partial collapse of the structure during ion-exchange [22]. This reduction in specific surface area and pore volume is accompanied by an increase in the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio (Table S2), indicating dealumination during ion exchange [23]. SEM image is given in Fig. S2, showing a porous structure with particle sizes in the range of $0.6\text{--}2.2\text{ }\mu\text{m}$.

The XRD spectra of Zn-Ni-HY/bentonite show the two distinct diffraction peaks at $2\theta = 37.2^\circ$ (♦) and 43.2° (♣), assigned to [101] plane

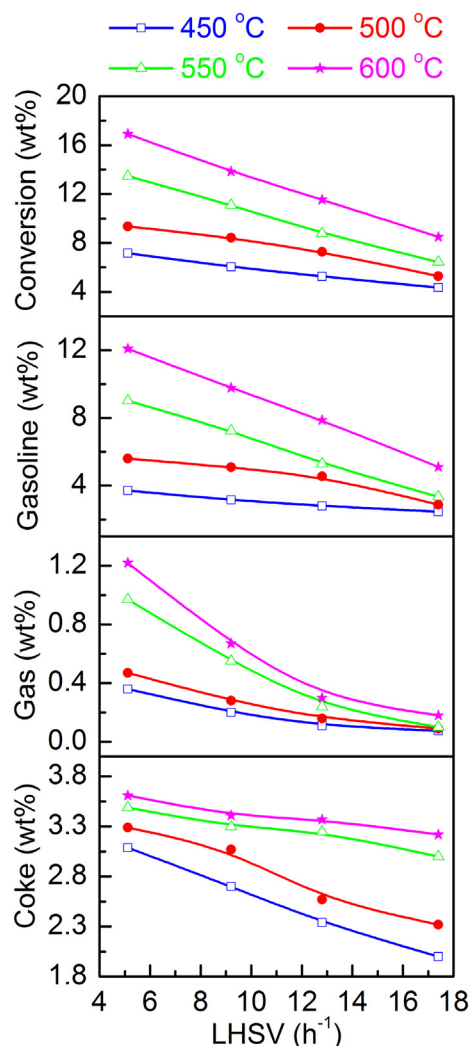


Fig. 3. The conversion of furfural extract, the yields of gasoline, gas and coke for catalytic conversion of furfural extract over HY/bentonite catalyst for TOS of 30 min under different temperature and liquid hourly space velocity.

of ZnO (JCPDS No.36-1451) and [200] plane of NiO (JCPDS No.47-1049), individually. The specific surface area and pore volume of Zn-Ni-HY/bentonite are $271\text{ m}^2\text{ g}^{-1}$ and $0.102\text{ cm}^3\text{ g}^{-1}$, which are comparable with those for HY/bentonite.

3.2. Catalytic conversion of the furfural extract

The catalytic reactions were performed in a bench scale fixed bed reactor using the in-house prepared HY/bentonite catalyst at various temperatures and liquid hourly space velocities. The yields of gasoline, gas products and coke are given in Fig. 3. It is evident that a reduction of the LHSV at constant temperature by reducing the feed rate leads to higher yields of gasoline, gaseous products and coke. This is attributed to the longer contact time between the feed and the active sites of the catalyst [24]. Similarly, when increasing the temperature at the same LHSV value, the conversion of furfural extract, the yields of gasoline products, gas product and coke increase [25]. A gasoline yield of 7.3 wt% was obtained at 550°C and LHSV of 9.2 h^{-1} . The conversion of the furfural extract to gasoline was relatively low (e.g., $< 18\text{ wt}\%$, see Fig. 3). This is likely due to the presence of highly condensed polycyclic aromatics, which are known to be difficult to crack, even in the presence of catalysts. In addition, it is low because the furfural conversion calculations are only based on amounts of gasoline, coke and gas products formed (Eq. 4), ignoring all other liquid products.

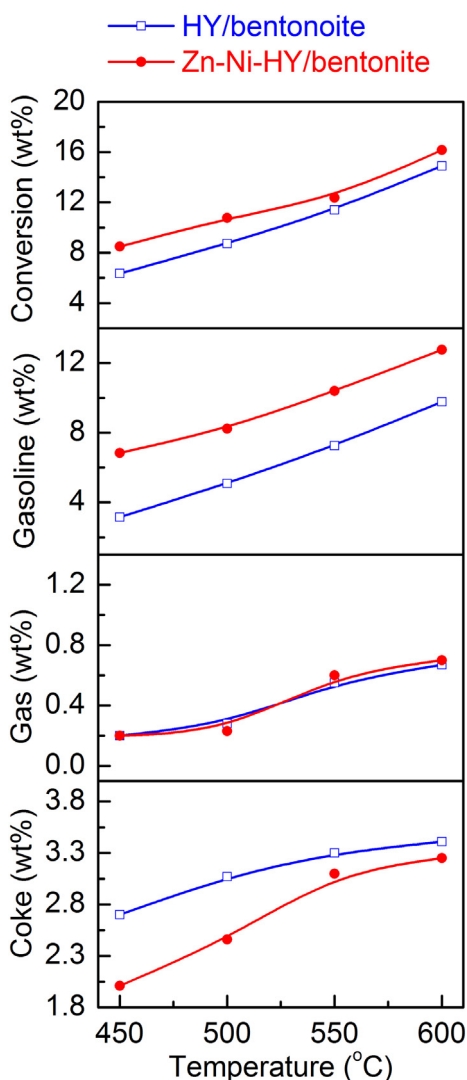


Fig. 4. The conversion of furfural extract, the yields of gasoline, gas and coke for catalytic conversion of furfural extract over HY/bentonite and Zn-Ni-HY/bentonite catalysts for TOS of 30 min and LHSV of 9.2 h⁻¹ under different reaction temperature.

The conversion of furfural extract was considerably enhanced when using the promoted Zn-Ni-HY/bentonite catalyst, see Fig. 4 for details (constant LHSV of 9.2 h⁻¹ and different reaction temperatures). The gasoline yield reached 10.4% at 550 °C and LHSV of 9.2 h⁻¹ over Zn-Ni-HY/bentonite catalyst, which is ca. 42% higher than that obtained over HY/bentonite catalyst. As such, the promotion of the catalyst with Ni and Zn has a positive effect which may be attributed an enhanced catalytic activity for C–C and C–H bond cleavage [26]. Interestingly, the coke yield over Zn-Ni-HY/bentonite catalyst is lower than that over

HY/bentonite catalyst, might be related to the decreased acidity of HY zeolite modified by the addition of Zn and Ni [27]. In addition, though not investigated here, the promoters also are known to lead to less structural damage at reaction conditions by reducing the rate of aluminum loss from the zeolite framework [28,29]. Though a remarkable improvement has been obtained over the promoted Zn-Ni-HY/bentonite catalyst, it still needs to be noted here that further development of more active catalysts is essentially needed to achieve a satisfied gasoline yield at the reaction conditions close to FCC process (e.g. contact time of few seconds).

3.3. Relevant properties of the gasoline fraction and other distillation fractions

The chemical composition of the representative isolated gasoline fractions obtained from catalytic conversion of the furfural extract over HY/bentonite and Zn-Ni-HY/bentonite catalysts (550 °C and a LHSV of 9.2 h⁻¹) is provided in Table 2, showing that the gasoline fraction contains relatively low amount of olefins and high amount of *i*-paraffins and aromatics. The research octane number (RON, calculated using Eq. 5, [30]) of the gasoline produced over HY/bentonite catalyst is 92.7. Compared with HY/bentonite catalyst, Zn-Ni-HY/bentonite catalyst produced more aromatics and less naphthenes, resulting in a higher RON of 93.7. These RONs are slightly higher than those for the regular and premium gasoline (e.g., 85 and 90, respectively in Iraq [31]). Thus the produced gasoline from catalytic conversion of furfural extract appears to have potential to be used as a base gasoline for regular and premium automobile gasoline production.

$$RON = \frac{C_{aromatics} + 191.9}{2.436} \quad (5)$$

Furthermore, the overall liquid products obtained from the experiments at 550 °C and a LHSV of 9.2 h⁻¹ using HY/bentonite and Zn-Ni-HY/bentonite catalysts were distilled to fractionate the petroleum products. The distilled fractions and the properties are listed in Table 3. The total yield of light naphtha, heavy naphtha, kerosene, gas oil, and lubricating oil produced over HY/bentonite catalyst is 77.6 wt% based on the total liquid products from catalytic conversion of furfural extract. Comparatively, the total yield of these distilled petroleum products is significantly increased to 90.5% when using Zn-Ni-HY/bentonite catalyst, while the characteristics of the individual fractions are hardly changed. The octane numbers of the produced light naphtha (87–88) and heavy naphtha (85–86) are similar to the required specifications [31]. However the sulfur contents (0.4 wt%) are high, which is related to the high sulfur content (4.5 wt%) in the furfural extract. It is thus suggested to perform a hydrodesulfurization (HDS) [32] step before using them as the components for the automobile gasoline. The obtained kerosene fraction has slightly higher smoke point (26–28 mm) than required (25 mm) and also needs an HDS treatment due to its relatively high sulfur content (0.5–0.6 wt%). The produced gas oil fraction has a higher diesel index (61–64) than the required (55) and has potential to be used as a light diesel fuel after HDS processing to reduce its sulfur content (1.1–1.2 wt%). The lubricating oil fraction has

Table 2

Chemical composition of the gasoline products from catalytic cracking of furfural extract over HY/bentonite and Zn-Ni-HY/bentonite catalysts.

Chemical composition	HY/bentonite catalyst		Zn-Ni-HY/bentonite catalyst		Commercial reference [31]
	Concentration (wt%)	Concentration (vol%)	Concentration (wt%)	Concentration (vol%)	
<i>n</i> -Paraffins	17.7	19.9	17.5	19.6	–
<i>i</i> -Paraffins	22.0	23.7	22.0	23.7	–
Olefins	10.2	10.8	10.1	10.6	18
Naphthenes	16.2	16.0	14.1	14.0	–
Aromatics	34.0	29.6	36.3	31.9	35
Total C ₁₄ ⁺	–	–	–	–	–

Table 3
Properties of the distilled petroleum products from catalytic cracking of furfural extract over HY/bentonite and over Zn-Ni-HY/bentonite catalysts.

	Characteristics	HY/bentonite catalyst	Zn-Ni-HY/bentonite catalyst	Commercial reference [31]
Light naphtha	Yield (wt%)	1.4	2.2	–
	Specific gravity (15.6 °C/15.6 °C)	0.708	0.701	0.7
	API gravity	68.4	70.4	70.6
	Sulfur content (wt%)	0.4	0.4	0.1
	Net heat (cal g ⁻¹)	10,477	–	11,000
	Octane number	87	88	85
Heavy naphtha	Yield (wt%)	6.4	8.1	–
	Specific gravity (15.6 °C/15.6 °C)	0.751	0.76	0.775
	API gravity	56.8	54.0	51
	Sulfur content (wt%)	0.4	0.4	0.1
	Net heat (cal g ⁻¹)	11,150	–	11,000
	Octane number	85	86	85
Kerosene	Yield (wt%)	8.4	12.6	–
	Specific gravity (15.6 °C/15.6 °C)	0.827	0.810	0.801
	API gravity	39.43	43.19	45.15
	Smoke point (mm)	28	26	25
	Sulfur content (wt%)	0.6	0.5	< 0.2
	Net heat (cal g ⁻¹)	10,846	–	10,900
Gas oil	Yield (wt%)	7.4	11.3	–
	Specific gravity (15.6 °C/15.6 °C)	0.848	0.850	0.85
	API gravity	35.42	35.42	34.97
	Sulfur content (wt%)	1.2	1.1	1.0
	Net heat (cal g ⁻¹)	10,707	–	10,800
	Aniline point (°C)	83	80	70
Lubricating oil	Diesel index	64	61	55
	Yield (wt%)	54.0	56.3	–
	Viscosity at 40 °C (cSt)	298.3	297.0	–
	Viscosity at 100 °C (cSt)	19.8	19.1	17–21
	Viscosity index (min)	71.6	72.0	95

a lower viscosity index (71.6–72.0) than required (95) and might be used as a blend for e heavy lubricating oils.

4. Conclusions

We have successfully demonstrated catalytic conversion of furfural extract in a fixed bed reactor using the shaped and promoted HY catalysts followed by the distillation to obtain various petroleum products, viz., light naphtha, heavy naphtha, kerosene, gas oil and a lubricating oil at various temperatures (T, 450–600 °C), 24 g of catalyst loading and liquid hourly space velocities (LHSV, 5.1–17.4 h⁻¹). Higher reaction temperatures and lower LHSVs favor the conversion of the furfural extract and the gasoline yield over HY/bentonite catalyst, which can be significantly enhanced by promoting the catalyst with Zn and Ni. The gasoline product from catalytic conversion of the furfural extract at temperature of 550 °C and LHSV of 9.2 h⁻¹ shows a high research octane number (92.7 over HY/bentonite catalyst vs. 93.7 over Zn-Ni-HY/bentonite catalyst) and has potential to be used as a basic gasoline for automobile gasoline production. The light and heavy naphtha, kerosene, gas oil, and lubricating oil fractions with high yield (ca. 77.6 wt% over HY/bentonite catalyst vs. 91.5 wt% over Zn-Ni-HY/bentonite catalyst) are of interest to be used as automobile gasoline, kerosene, light diesel fuel, and as a blend for heavy lubricating oils. The modification of HY zeolite by Zn and Ni shows significant enhancement on catalytic activity with respect to the yield and RON of gasoline, and the yield of the overall distilled petroleum products as well.

Declaration of Competing Interest

The authors declare no competing interests.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.catcom.2019.105834>.

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