Synergistic effect with and without Ni metal over ZSM-5/SAPO-11 catalyst in hydrodeoxygenation of palm oil

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Abstract. Hydrodeoxygenation (HDO) of palm oil by catalytic for biofuels has attracted the researchers in the recent years. Currently available catalysts are typically intensive on sulfided CoMo and NiMo catalysts, which certainly modeled by the sulfur contamination in the final liquid product. Consequently, exploring the non-sulfured catalyst is still vulnerable challenge and has ultimate importance. In this paper, sulfur free catalyst with and without Ni based SAPO-11/HZSM-5 is synthesized according to incipient wetness impregnation method. The prepared catalyst was tested by performing HDO of palm oil for product selectivity. Different analytical techniques were used like: XRD, FESEM, Pyridine-FTIR and H2-TPD to explore the physicochemical properties of the novel catalyst. In addition, Ni supported ZSM-5/SAPO-11 catalyst with medium acidity showed greater properties because of the synergistic effect of Ni metal and acidic support than the catalyst without Ni support. The results showed that the, catalytic acidity has suggestively inclined the catalytic performance and product selectivity. The maximum selectivity of biofuel was achieved with ZSM-5/SAPO-11 over Ni support under the mild reaction condition at 350°C is indicating its auspicious part in this reaction. The acquired results determined that the metal and acid sites showed crucial role for the development of an effective catalyst for HDO of palm oil over Ni supported catalyst.

Keywords: Catalyst, HDO, Selectivity, Bio-fuels.

1. Introduction

Increasing industrialization and transportation instigated depletion of fossil fuels worldwide which increased the interest of researchers to explore the renewable energy sources in order to fulfill the future energy demand. From the literature it has been obvious that, the primary energy consumption of transport sector is about 40% [1]. Besides, during 1990 to 2011 combustion of fossil fuels by road transportation sector, sea ships and airplane increased the gas emissions about 52 to 80% [2]. Therefore, it is essential to explore the renewable, sustainable and effective energy source with probably lesser emissions. In this regard bio-fuels from vegetable oils showed potential in the manufacture of liquid fuels [1] since, high productivity of plants as oil per hectare, such as palm trees can be counted as the most promising and viable source of feedstock [2]. Current information have shown that, the hyd?roprocessing is well-known method used in petroleum refineries to crack larger hydrocarbons like fatty materials to obtain alkane like hydrocarbon and to eliminate the Sulphur content [3]. Moreover, hydproprocessing is comprising on dehydration, which removes H2O molecules and establishing hydrocarbons with equivalent number of carbon atoms similar to original fatty acid. Reaction (1); decarboxylation, which removes CO2 – Reaction (2); and decarboxylation, which removes CO -
Reaction (3). Throughout the decarboxylation and decarbonylation reaction, the hydrocarbons that are formed have one less carbon atom than the precursor fatty acid in presence of suitable catalyst [2].

\[
\begin{align*}
\text{R-COOH} + 3 \text{H}_2 & \rightarrow \text{RCH}_3 + 2\text{H}_2\text{O} \quad (1) \\
\text{R-COOH} & \rightarrow \text{RH} + \text{CO}_2 \quad (2) \\
\text{R-COOH} + \text{H}_2 & \rightarrow \text{RH} + \text{CO} + \text{H}_2\text{O} \quad (3) \\
\text{CnH}_2\text{nO}_2 + 3\text{H}_2 & \rightarrow \text{CnH}_2\text{n} + 2 + 2\text{H}_2\text{O} \quad (4)
\end{align*}
\]

The leading catalyst for HDO is mostly centered on noble metals such as Pt, Pd, and transition metals NiMo, CoMo, and NiW on the support of \( \gamma \text{-Al}_2\text{O}_3 \), activation carbon, SiO\(_2\), and SBA-15 etc. Additionally, noble metals presented stability and high activity, but their high cost limited their vital application on a large scale [4]. Sulfided catalyst Mo and W were modified by Ni and Co and used in HDO of vegetable oil and exhibited acceptable hydrocarbon yield with good stability but inevitably suffered from sulfur contamination in the final products [5]. Therefore, a novel catalyst system is need to be developed to decrease the contents of sulfur and olefins from gasoline [6] in order to improve the selectivity of alkane in aviation fuel.

Obviously, it is anticipated that this type of catalyst arrangement can be attained by joining or linking dissimilar zeolites with specific ratio. It is generally known that zeolite ZSM-5 has exceptional activity for equally HDS and light alkane and alkene aromatization. Besides, silicoaluminophosphate zeolite SAPO-11 is broadly used for isomerization of \( \text{C}_8\text{-C}_{15} \) hydrocarbons for gasoline production and for dewaxing process. The type of support is of central rank to obtain highly dispersed Ni particles and catalysts with excellent performance [7]. It has been also recognized that Ni particle loading by suitable supports such as metal oxides, zeolites, mesoporous silica and nanocars possibly increase the catalytic activity of Ni-based catalysts attributed to the alteration of many properties (such as Ni particle size distribution, reducibility, and metal-support interactions). Certainly, catalytic action and its constancy is sturdily depending on both structure of support and interaction between metal and support [25,26]. Therefore, modified transition metals with Ni showed high catalytic performance in HDO reaction.

Certainly, it is important to consider a catalyst arrangement comprising on a ZSM-5/SAPO-11 composite material with suitable configuration of Ni support in order to provide desulfurization and product selectivity. Furthermore, the composites of zeolites described in the literature are consisted on two or more individual zeolites mixed mechanically or synthesized. The mechanical mixing might hinder the intimate contacts of different zeolites and thereby reduce their synergism. For this purpose, Due to this fact a sequence of aluminosilicate/(silico) aluminophosphate ZSM-5/SAPO-11 composites were synthesized with and without Ni support by an impregnation method. After catalyst preparation, catalytic performance was investigated for HDO of palm oil to hydrocarbon fuel. Different analytical techniques were used to characterize the catalyst corresponding: X-ray diffraction XRD, Field Emission Scanning Electron Microscopy (FESEM), infrared spectroscopy of adsorbed pyridine (Pyridine-FTIR) and \( \text{H}_2\)-TPD to investigate the physicochemical properties of the novel catalyst.

2. Materials and Methodology

2.1 Materials

ZSM-5 and SAPO-11 were commercially purchased from Zeolite International. Analytic graded Ni(NO\(_3\))\(_2\cdot 6\text{H}_2\text{O}\) (crystallized, \( \geq 97\% \)) and n-Heptane \( \geq 99\% \) were purchased from Merck. Purified \( \text{H}_2\), \( \text{N}_2\), He and gas mixtures 5% \( \text{H}_2\) in \( \text{N}_2\) were obtained from gas link Malaysia Sdn. Bhd.

2.2. Synthesis of Catalyst
Primarily without Ni support catalyst ZSM-5 and SAPO-11 were prepared by impregnation method. First of all, 50% amount of ZSM-5 and 50% SAPO-11 was mixed in de-ionized water at 60°C for 8 hours under vigorous agitation was set to 800 rpm until a homogenous mixture was obtained. After impregnation, the catalyst was left for 2 h at room temperature, then dried in an oven at 100°C for 12 h to evaporating the excess water, moisture and finally calcined in air at 550°C (with heating rate of 3°C/min) overnight. Besides, Ni based support catalysts was synthesized by wet-impregnating method. The catalyst support was retained in an aqueous solution of Ni(NO₃)₂·6H₂O as Ni precursor with the desired 10 wt.% Ni loading. After impregnation, the catalyst was left for 2 h at room temperature, then dried in an electric oven at 100°C for 12 h to evaporating the excess water and then finally calcined in ambient condition at 550°C (with heating rate of 3°C/min) overnight. Additionally, prior to the HDO reaction, catalyst was reduced in hydrogen environment with 35 bars pressure at 450°C for 2 h.

2.3. Characterization of catalyst

Aiming to find out the characteristics of synthesized catalyst, X-ray diffraction (XRD) patterns were attained by P Analytical X' Pert diffractometer with Cu Kα radiation is used due to line is about 90× more concentrated than the white radiation of a similar wavelength. Therefore, white radiation from a copper anode is too weak to be of any practical use for powder diffraction in the laboratory. The working voltage and current of X-ray tube were 40 kV and 40 mA, respectively. Surface morphology of catalysts were examined using a field emission scanning electron microscope (FESEM), ZEISS Gemini SEM 300 equipment. The catalyst samples were positioned on a conductive carbon tape adhered to an aluminum sample holder. The nature of the surface Bronsted and Lewis acid sites were determined by FTIR analyses of pyridine adsorbed on samples (Py-FTIR) was done on a Bruker FTIR IFS 66/S instrument at intensities 4000–500 cm⁻¹. H₂-Temperature-programmed reduction (H₂-TPD) is a extensively used tool for the classification of metal oxides, mixed metal oxides, and metal oxides dispersed on a support. 200 mg of individually every sample was loaded in TPD cell then purged with He gas at 170°C for 30 min at a flow rate of 20 ml/min. After sample cleaning process, each sample was reduced with mixed gas of 5%H₂/95% with N₂ flowrate of 20 ml/min at 900°C for 1 h. Once the TCD (thermal conductivity detector) signal become constant, H₂-TPD was performed with a heating rate of 10°C/min, formerly desorbed hydrogen was recorded by means of TCD signal.

2.4 Catalytic Activity Studies

The experimental setup used for this study is provided by Amar Equipment Private Limited, Mumbai, India. The stainless-steel autoclave (SS 316) batch react of 1 L was used to carry out the HDO experiments. The reactor comprised on electric furnace for heating. The reactor was equipped with thermocouple, pressure gauge, safety valve, stirrer, and vent valve. The complete experimental setup is shown in Fig. 1. For each experiment 2g of prepared catalyst was loaded in the autoclave reactor and purged with N₂ for 5 min and then filled with pure H₂ at initial pressure of 25 bars at room temperature and reduced at 450°C for 2h. Then reactor was cooled with internal cooling coil system and water as cooling medium. The H₂ gas was released from the reactor in the atmosphere and then mixture of Palm oil and n-heptane with 10:90 ratio was injected from top inlet in auto clave batch reactor through funnel. Same procedure was repeated each HDO experiment to purge and load the H₂ gas for. The reaction was performed at hydrogen pressure of 25 bar with stirring speed of 500 rpm for 2h. The autoclave was heated electrically to reach the temperature of 350°C measured by K-type thermocouple. Once the reactor temperature reached to set temperature, the reactor pressure reached to 65 bars and then decreased to 58 and 55 bars, correspondingly due to the catalysts activity and enhanced the hydrogenation reaction which lead hydrogen consumption till the HDO reaction completed.
Afterward completion of HDO reaction, autoclave was cooled to the ambient temperature with water cooling coil. The gaseous products were not collected because of restrictions in the experimental setup design and vented out slowly through the bubbler to avoid any contamination in the adjacent environment. At the end autoclave was open and liquid along with catalyst was withdrawn and catalyst was separated from liquid product by vacuum filtration process. After separation of liquid product was stored in glass bottle and then analyzed through CHNS and GC-MS.

The yields of liquefied products were calculated by using Eq.(5).

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\text{Yield(\%)} = \frac{\text{Weight of product} \times \text{Weight of liquid product}}{100}
\]

All the experimental sets were replicated 3 times under the same settings to avoid from any error and authenticate the experimental results.

3. Results and discussion

For characterization of catalyst the following analysis were performed such as X-ray diffraction XRD, Field Emission Scanning Electron Microscopy (FESEM), Fourier transformed infrared spectroscopy of adsorbed pyridine (Pyridine-FTIR) and H₂-TPD. The detail of each analysis is given in the sub section.

3.1 Characterization of catalyst

3.1.1 X-ray diffraction (XRD) analysis of catalyst

The XRD patterns of ZSM-5/SAPO-11 and Ni/ ZSM-5/SAPO-11are shown in Fig 2 (a) and (b). Diffraction peaks of ZSM-5/SAPO-11 and Ni/ ZSM-5/SAPO-11 at 2θ=1–2° and 50° were observed. The
XRD patterns of the micro and mesoporous ZSM-5/SAPO-11 samples showed the typical low XRD angle ZSM-5/SAPO-11 in the range 2θ = 1–2° due to the micro and mesoporous structure zeolite peaks in wide angle. However, Fig. 2(b) the Ni supported ZSM-5/SAPO-11 sample presented a broad mesoporous lump and sharp crystalline zeolite peaks as a result of the structure directing of nickel ions for tetrahedrally linking aluminosilicate/silicoalumino phosphate framework. The strength of diffraction peaks for the Ni/ ZSM-5/SAPO-11 catalysts increased significantly in comparison to that of ZSM-5/SAPO-11. A small decline of crystallinity was detected when nickel was loaded. This was probably caused by high dispersion of Ni species on ZSM-5/SAPO-11 surface, might be due to the effect of Ni species was diffused into the microchannels of ZSM-5/SAPO-11 and no diffraction peaks of NiO crystals (2θ=37.2, 43.5°) were observed in catalyst is assigning to high dispersion of NiO crystals in the structure of ZSM-5/SAPO-11 [8].

3.1.2 Field Emission Scanning Electron Microscopy (FESEM) analysis of catalysts

The FESEM images of the catalysts are presented in Fig. 3a and b. According to Fig. 3a the ZSM-5/SAPO-11 phase is existing in form of pseudo-spherical masses of the size fluctuating from 7 to 8 mm involving uniform cubic plate monocrystals 0.5 mm in size [6]. The ZSM-5/SAPO-11 existing with a comparatively flat surface with the size of many micrometers. Fig. 3 (b) Ni/ZSM-5/SAPO-11 showed composite with a fairly distinct morphology, compared with the catalyst as revealed in Figure 3(a). The composite with Ni/ZSM-5/SAPO-11 showed the sphere-like particles ranging from 2.3 to 4.8 μm diameter. However, Ni/SAPO-11, the support size persisted virtually unaffected and the size of the NiO elements remained between 30–100 nm. This effect is because of the leading pore filling effect of Ni at lower loading and the start of SAPO-11/ZSM-5 deposition on catalyst surface at moderate loading [4].

3.1.3 FTIR and Pyridine FTIR analysis of catalyst

Fig. 4 displays Py-IR spectra of the catalysts tested at an ambient temperature to examine the catalysts acidity. Figure 4 (a) depicted that, ZSM-5/SAPO-11 catalyst exhibited several peaks due to strong Lewis
acidic site-bonded pyridine at 850 cm\(^{-1}\), weak Lewis acidic site-bonded pyridine at 1450 cm\(^{-1}\) that are representative of pyridinium ions at surface Brønsted acid sites (PyH\(^+\)). While Figure 4 (b) Ni/ZSM-5/SAPO-11 catalyst, the Bronsted acid sites were slightly reduced and Lewis acid sites were largely improved. Furthermore, bands at 750-850 cm\(^{-1}\) and 950-1438 cm\(^{-1}\) are credited to the adsorption of pyridine corresponding on Lewis acid sites formed by metallic Ni along with the support [9]. The band appearing at 2950-3600 cm\(^{-1}\) is attributed to the vibration frequency of the pyridine ring on Bronsted acid sites formed by both Ni and the support. From the results obtained by Py-IR for a series of catalyst samples, it can be indicated that the Ni improved the amount of Lewis and Bronsted acid sites of catalyst (Ni/ZSM-5/SAPO-11) in relation to without Ni zeolite (ZSM-5/SAPO-11). In light of existing consequences further proves the contact between SAPO-11 and ZSM-5 crystals in the composite is in line with XRD results. The above observations reveal that developed catalysts exhibited considerable both Bronsted and Lewis acid sites are existing on these catalysts in which one or both may be accountable for the hydrodeoxygenation of palm oil and the formation of jet fuel hydrocarbons as discussed in the catalytic activity part [10].

3.1.4 \(\text{H}_2\)-Temperature-programmed reduction (\(\text{H}_2\)-TPD) analysis of catalyst

Fig. 5 represents the \(\text{H}_2\)-TPD profile of catalyst without and with addition of Ni metal with ZSM-5/SAPO-11 zeolites.

![Graph showing H2-TPD profiles of ZSM-5/SAPO-11 with and without Ni nanoparticles.](image)

Fig. 5 shows the without Ni metal ZSM-5/SAPO-11 peak at high temperature from 100 to 700°C showed no peak which is assigned to absence of Ni metal in the catalyst. However, Ni metal with ZSM-5/SAPO-11 pure NiO showed one lower and higher reduction peak in temperature range of 200-300 and 300-600°C, correspondingly signifying the coexistence of small and large Ni nanoparticles. The peak centered at temperature from 300 to 600°C matches to the dispersed Ni species with ZSM-5/SAPO-11, which has solid link with support. Therefore, \(\text{H}_2\)-TPD of Ni/ZSM-5/SAPO-11 and without Ni metal established the coexistence of NiO, nickel aluminate spinel and framework-structured nickel in the materials [8]. Previous study revealed that, Ni with oxide support contained on free NiO particles and the interactional Ni\(_{2+}\) specie was covalently joined with support by linking O\(_2\) atom and leading in reduction of Ni\(_{2+}\) species at higher temperature.
In the current case Ni$^{2+}$ species with less loadings might exceedingly scatter on the surface of prepared ZSM-5/SAPO-11. Also, directly joining to the support and barely declined by pure H$_2$ which bringing about the drop-in peaks located at higher temperature. However, bulkier NiO particles can be formed at the incremental Ni loadings, thus shifting the peaks at reduced temperature. Therefore, Ni$^{2+}$ species forcefully interacting with ZSM-5/SAPO-11 and joining to fix the metallic Ni particles in HDO of palm oil [4]. These outcomes signifying that in all prepared catalysts, NiO is predominantly placed at the outside of zeolite is evidently specified by TPD analysis.

4. Characterization of liquid product

4.1. Elemental analysis of jet fuel product

Table 1: Elemental analysis of jet fuel produced with ZSM-5/SAPO-11 and Ni/ZSM-5/SAPO-11.

| Elemental analysis | ZSM-5/SAPO-11 | Ni/ZSM-5/SAPO-11 |
|-------------------|---------------|------------------|
| C                 | 65.34         | 76.82            |
| H                 | 12.18         | 12.36            |
| N                 | 6.75          | 9.46             |
| S                 | 0.92          | 0.92             |
| O (by difference) | 14.99         | 0.44             |
| HHV (MJ/kg)       | 36.96         | 43.70            |

The content of oxygen measured was in all the both catalyst was very low and comparable with the amount found in commercial jet fuels. Results also showed that the quality of jet fuel was HHV was improved from 65.35 to 76.82 (MJ/kg), while the concentration of hydrocarbon compounds was also increased with addition of Ni metal Ni/ZSM-5/SAPO-11 catalyst therefore, synergistic effect was observed. From this study, the performance of Ni based catalyst in-dicating that the jet fuel produced have equivalent calorific value with the commercial jet fuel (43.71MJ/kg) [11].

4.2. GC/MS analysis of jet fuel after HDO of TGs using ZSM-5/SAPO-11 and Ni/ZSM-5/SAPO-11 catalyst

The catalytic activity of ZSM-5/SAPO-11 and Ni/ZSM-5/SAPO-11 catalyst for HDO of palm oil was investigated at 350°C and 600 rpm. The main compounds after HDO of TGs to jet fuel were identified by GC/MS analysis are summarized in Table 2.

Table 2: GC/MS analysis after HDO of TGs by ZSM-5/SAPO-11 and Ni/ZSM-5/SAPO-11 catalyst.

| Compounds Identified | ZSM-5/SAPO-11 | Ni/ZSM-5/SAPO-11 |
|----------------------|---------------|------------------|
| n-Alkane (C$_8$ – C$_{15}$) | 4.87          | 8.15             |
| n-Alkane (C$_{16}$ – C$_{18}$) | 35.75         | 67.52            |
| Olefins              | 20.51         | 6.32             |
| Aromatic             | 3.46          | 15.97            |
| Carboxylic acid      | 22.68         | 2.04             |
| Ketone               | 10.31         | ND               |
| Alcohol              | 1.099         | ND               |
| Distearyl sulfide    | 1.289         | ND               |
| Total Conversion (%) | 100           | 100              |

ND = Not detected.
From GC/MS analysis the major compounds were observed n-Alkane from (C₁₆–C₁₈) over ZSM-5/SAPO-11 and Ni/ZSM-5/SAPO-11 with 35.75% and 67.52%, respectively. Results without Ni loading indicated less catalytic activity, selectivity and product yield when ZSM-5/SAPO-11 was used. In addition, the mesoporous system with Ni metal improved the catalytic efficiency [1]. Therefore, reduction in selectivity can be associated to the absence of NiO particles and the interactional Ni₂⁺ at moderate temperature. On the other hand, Ni addition with Ni/ZSM-5/SAPO-11 catalyst exhibited much higher activity than without Ni in ZSM-5/SAPO-11, signifying the core active sites of Ni species which shows the highest catalytic action. Such an exceptional catalytic stability of Ni/ZSM-5/SAPO-11 is credited to the exceedingly stable Ni nano-particles attached to the zeolite support [12].

After subsequent aromatization alkenes were changed to aromatics which resulted a decrease in carboxylic acid from (22.68 to 2.04%) through decarboxylation reaction. As a result, a desirable mixture of linear hydrocarbons (67.52 %), and aromatics (15.97 %) within the aviation fuel range (C₁₀–C₄₀) was obtained. On the other side, n-alkanes from (C₈–C₁₈) with (4.87%), n-alkanes from (C₁₆–C₁₈) with (35.75 %) and carboxylic acid (22.68 %) and ketone with (10.31%) were observed. However, some oxygenated compounds such as carboxylic acid with (22.68 %) were also observed when cracking was performed only with ZSM-5/SAPO-11zeolite. Actually, these compounds are resulted from incomplete deoxygenation reaction due to weak acid sites due to absence of Ni metal that is in line with our pyridine FTIR results. Besides the hydrogenation and hydro cracking of Palm oil over Ni/ZSM-5/SAPO-11 more alkanes and less quantity of carboxyl group was observed while, no ketenes, alcohol and distearyl sulfide were detected. However, Ni/ZSM-5/SAPO-11 resulted increasing dehydration trend, which improved the jet fuel range hydrocarbons yield from (C₈–C₁₄) and (C₁₆–C₁₈) was obtained.

5. CONCLUSIONS

A sequence of ZSM-5/SAPO-11 and Ni/ZSM-5/SAPO-11 catalysts were fashioned by wetness impregnation technique and analyzed by XRD, FESEM, Pyridine FTIR, H₂-TPD. Among the catalysts Ni/ZSM-5/SAPO-11 exhibited the maximum catalytic activity. The XRD pattern of Ni/ZSM-5/SAPO-11 show highly crystalline in contrast to ZSM-5/SAPO-11 with no significant presence of impurities. In addition, Ni/SAPO-11/ZSM-5 micrograph showed little Ni particles deposited on the surface of catalyst. It should be noticed that the no any reduction peak at the highest temperature (>900oC) was observed over ZSM-5/SAPO-11 framework. However, Ni/ZSM-5/SAPO-11 shows only one major reduction peak between 300–600°C was observed which suggests the pure NiO coexistence of small and large Ni particles ZSM-5/SAPO-11 catalyst exhibited several peaks due to strong Lewis acidic site-bonded pyridine at 850cm⁻¹, weak Lewis acidic site-bonded pyridine at 1450 cm⁻¹ that are representative of pyridinium ions at surface Bronsted acid sites (Py H⁺). While, Ni/ZSM-5/SAPO-11 encompasses two types of weak and strong acid sites. Ni/ZSM-5/SAPO-11 catalyst, the Bronsted acid sites were slightly reduced and Lewis acid sites were largely improved. The bands at 750-850 cm⁻¹ and 1438 cm⁻¹ credited to the adsorption of pyridine corresponding on Lewis acid sites formed by metallic Ni as well as the support. The hydrogenation, hydrodeoxygenation and catalytic cracking of Palm oil appears to deliver an efficient pathway for bio aviation fuel. In addition, a significant extent of liquid fuel from (60–77%) were correspondingly attained. Additionally, required mixture of linear hydrocarbons with (43.2 wt%), aromatics (6.3 wt%), and cycloalkanes (28.1 wt%) within the aviation fuel range (C₈–C₁₄) is also noticed. The jet fuel produced by Ni based catalyst have synergistic effect and comparable calorific value of 43.70 that with the commercial jet fuel.
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