Biodiesel Synthesis Catalyzed by Transition Metal Oxides: Ferric-Manganese Doped Tungstated/ Molybdena Nanoparticle Catalyst

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Abstract: The solid acid Ferric-manganese doped tungstated/molybdena nanoparticle catalyst was prepared via impregnation reaction followed by calcination at 600°C for 3 h. The characterization was done using X-ray diffraction (XRD), Raman spectroscopy, thermal gravimetric analysis (TGA), temperature programmed desorption of NH₃ (TPD-NH₃), X-ray fluorescence (XRF), Transmission electron microscope (TEM) and Brunner-Emmett-Teller surface area measurement (BET). Moreover, dependence of biodiesel yield on the reaction variables such as the reaction temperature, catalyst loading, as well as molar ratio of methanol/oil and reusability were also appraised. The catalyst was reused six times without any loss in activity with maximum yield of 92.3% ± 1.12 achieved in the optimized conditions of reaction temperature of 200°C; stirring speed of 600 rpm, 1:25 molar ratio of oil to alcohol, 6 % w/w catalyst loading as well as 8 h as time of the reaction. The fuel properties of WCOME’s were evaluated, including the density, kinematic viscosity, pour point, cloud point and flash point whereas all properties were compared with the limits in the ASTM D6751 standard.

Key words: ferric-manganese doped tungstated/molybdena (FMWMo), WCOME’s (Waste Cooking Oil Methyl Esters), crystallite size, Debye-Scherrer’s relationship

1 INTRODUCTION

Ecological, financial and political attentions had marked the manufacture of bio-source energies extremely beneficial. Triglyceride based-diesel is attracted specific consideration since it would be synthesize employing a range of edible, non-edible plant oils and spent cooking oil and has been directly applied in present vehicles. The use of low-priced waste cooking oil and other acidic oils, permits for enhancing the synthesis and development of cost-effective view of low price based-biodiesel. Nonetheless, the acidic oils as raw materials have been incompatible for the present liquid alkaline catalysts. Therefore, a need for development of new catalysis system to overcome the technical issues associated with employment of homogeneous catalysis system is being very essential and accordingly researchers keep on developing a novel catalysis system which contributes positively on the budget of biodiesel industry. For example, the result of this development on the catalysis system it comes out with the Lewis-Bronsted heterogeneous catalysis system, which has a capability to prepare the low priced based-biodiesel simultaneously, involving physically powerful acidic heterogeneous catalysts, sulphated zirconium oxide, and recently ferric hydrogen sulphate, saccharides, organosulfonic functionalized mesoporous silica, WO₃/ZrO₂-Al₂O₃, zirconia MoO₃, supported tungsten oxide and TiO₂/SO₄²⁻/SnO₂, SO₄²⁻/SnO₂–SiO₂, SnO₂/SiO₂ and WO₃/MCM-14. Generally, almost all of the earlier investigation employing acidic solid catalysts determined the results of esterification process of acidic oils and little has been done on transformation of triglyceride utilizing acid catalysts for the synthesis of acidic oil based-biodiesel. The advantage of acidic heterogeneous catalysts attributes to their ability on producing highest yield of biodiesel via single step process using low price raw materials. However, the produced catalyst contain large density of acid sites compare to other catalysts and can easily undergo simultaneous esterification of free fatty acid and transterification of triglyceride i.e. produc-

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ing biodiesel in single step which can contribute to reduce the total cost of biodiesel.

A chief goal of this work is to evaluate catalytic performance of ferric-manganese doped tungstated/molybdena solid acid catalyst for biodiesel-forming type reactions. The FMWMo is synthesized by impregnation technique and categorized utilizing XRD, Raman spectroscopy, TPD-NH3, TGA, BET, XRF and TEM. In addition, the impact of methanolysis process variables has been also assessed.

2 EXPERIMENTAL

2.1 Material

Sample of waste cooking oil was obtained from a local market around Universiti Putra Malaysia. A total of 5 kg of oil was used for the optimization of biodiesel production process. The materials Ferric nitrate nonahydrate, manganese nitrate tetra hydrate, ammonium metatungstate hexahydrate(99.0%) as well as ammonium molybdate tetrahydrate were obtained from Sigma- Aldrich–Malaysia and used in the preparation of the catalysts.

2.2 Pre-treatment of Waste Cooking Oil

For a successful simultaneous reaction and high yield of biodiesel, the oil must be free of water and other impurities. Initially, the waste cooking oil was heated at temperature in range of 100-108°C to get rid of water by loss and food debris through filtration. The free fatty acid (FFA) content was determined by a standard titration method\textsuperscript{10}.

2.3 Characterization of Waste Cooking Oil

Determination of acid and saponification values of the waste cooking oil was carried out according to the standard AOCS methods\textsuperscript{11}.

2.4 Catalyst Preparation

To prepare the catalyst, Fe(NO\textsubscript{3})\textsubscript{3}·9H\textsubscript{2}O, Mn(NO\textsubscript{3})\textsubscript{2}·4H\textsubscript{2}O and ammonium metatungstate hexahydrate were dissolved in 200 ml of deionised water in amounts corresponding to the desired stoichiometry and the solution was added drop wise under vigorous stirring into 10% ammonium molybdate tetrahydrate solution. The mixture was left under vigorous stirring at room temperature for 4 h and then dried at 120°C calcined at 600°C for 3 h\textsuperscript{12}, and without the addition of Fe(NO\textsubscript{3})\textsubscript{3}·9H\textsubscript{2}O, Mn(NO\textsubscript{3})\textsubscript{2}·4H\textsubscript{2}O the same method was employed for the preparation of tungstated molybdena catalyst.

2.5 Catalyst Characterization

The powder X-ray diffraction analysis was carried out using a Shimadzu diffractometer model XRD 6000. The diffractometer, employing Cu-K\textsubscript{α} radiation, was used to generate diffraction patterns from powder crystalline samples at ambient temperature. The Cu-K\textsubscript{α} radiation was generated by using the Philips glass diffraction X-ray tube broad focus 2.7 kW type. The crystallite size D of the samples was calculated using the Debye-Scherrer’s relationship\textsuperscript{13,14}.

\[
D = 0.9 \lambda / (B \cos \theta)
\]

where D is the crystallite size, \(\lambda\) is the incident X-ray wavelength, \(B\) is the full width at half-maximum, and \(\theta\) is the diffraction angle.

The TGA analysis was carried out on a Mettler Toledo TG-SDTA apparatus(Pt crucibles, Pt/Pt– Rh thermocouple), with the purge gas (nitrogen) flow rate of 30 ml min\textsuperscript{−1} and the heating rate of 10°C min\textsuperscript{−1} from room temperature to 1000°C.

The acidity of the catalyst was studied by temperature programmed desorption using NH\textsubscript{3} as probe molecule. The TPD–NH\textsubscript{3} experiment was performed using a Thermo Finnigan TPDRO 1100 apparatus equipped with a thermal conductivity detector. Approx 0.1 g of catalyst was placed in the reactor, treated under 150°C for 15 min in N\textsubscript{2}(20 ml min\textsuperscript{−1}). 10% NH\textsubscript{3} in helium gas was ramped at 1°C min\textsuperscript{−1} for 60 min. The purging with N\textsubscript{2} was done at room temperature for 45 min to remove NH\textsubscript{3} in the gas phase. Then, the analysis of the NH\textsubscript{3} desorption was carried out between 50°C and 900°C under helium flow(15°C min\textsuperscript{−1}, 20 ml min\textsuperscript{−1}) and detected by using a thermal conductivity detector.

Meanwhile the elemental chemical composition of the prepared Ferric- manganese doped tungstated molybdena acid catalyst was determined by Shimadzu X-ray fluorescence (XRF) with a Rayny EDX-720 spectrometer.

TEM (Hitachi H-7100, Japan) was also used to examine the crystal shape. For TEM analysis, the powder was dispersed in deionized water, dropped onto carbon-cover copper grids placed on a filter paper and dried at room temperature.

The total surface area of the catalysts was done using Brunauer–Emmer–Teller (BET) method with nitrogen adsorption at −196°C. Analysis was conducted using a Thermo Fisher Scientific S.p.A (model: SURFER ANALYZER) nitrogen adsorption-desorption analyzer.

The Raman spectrum of the calcined FMWMo sample was obtained at room temperature in an air atmosphere. Raman measurements were done in Raman systems(R-2001TM) equipped with diode laser source operates at full power at 785 nm with a 500 m W output power. The spectral range 100-1200 cm\textsuperscript{−1} was studied. The instrument was calibrated using a standard isopropyl alcohol reference(783 nm). GRAMS/32AI (6.00) software (Galactic Inc.) was used to analyze all spectra.

2.6 Simultaneous Esterification and Transesterification Process

Simultaneous esterification and transesterification of waste cooking oil containing 17.5% free fatty acids were
carried out using the prepared Ferric-manganese doped tungstated molybdena acid catalyst in the BERGHOF high pressure laboratory reactor. A certain amount of catalyst, methanol and oil were added to the reactor. The reaction was started by stirring it at 600 rpm and heated to specified temperature and time. The reaction mixture was then cooled to room temperature and separated by centrifuge (Kubota 4200) at rpm 350 for 20 min.

2.7 Product Analysis

The analysis of the Waste Cooking Oil Methyl Esters (WCOME’s) contents in the sample was carried out through gas chromatography (GC) by using the Shimadzu GC-14B with flame ionization detector (FID), equipped with RtX-65 capillary column. Helium was used as carrier gas with the initial oven temperature at 100°C held for 0.5 min and subsequently increased to 280°C (hold 10 min) at 20°C min⁻¹. The temperatures of the injector and detector were set at 250°C and 280°C, respectively. In each run, 1 μL of the sample was injected into the column. Methyl heptadecanoate was used as an internal standard. The yield of WCOME’s was calculated by the following equation: 

\[
\text{Yield of WCOME’s} (%) = \frac{\text{Total weight of methyl esters} \times 100}{\text{Total weight of oil used}}
\]

2.8 Catalyst Reusability and Leaching Tests

The catalyst, separated from the reaction mixture through centrifuge at 350 rpm for 20 min. and then filtration, was initially washed with hexane to remove non-polar compounds such as methyl esters on the surface. Moreover, the catalyst was washed with methanol to remove polar compounds such as glycerol and finally dried at 120°C for 6 h. The leaching of the catalyst into the reaction mixture was determined by Shimadzu X-ray fluorescence (XRF) with a Rayny EDX-720 spectrometer.

2.9 Fuel Properties

The fuel properties of the WCOME’s (Waste Cooking Oil Methyl Esters) were appraised using the subsequent ASTM methods: cloud point (ASTM D2500), pour point (ASTM D97), kinematic viscosity (ASTM D445), flash point (ASTM D93) and density (ASTM D5002). Triplicate determinations were carried out for every experiment and the data are presented as mean ± standard deviation.

3 RESULTS AND DISCUSSION

3.1 Characterization of Waste Cooking Oil

The saponification and acid value of waste cooking oil were found to be 173 mg KOH/g and 35 mg KOH/g, respectively, corresponding to a free fatty acid (FFA) level of 17.5%.

3.2 Biodiesel Determination through GC Analysis

The GC results have indicated that WCOME’s showed the lowest percentage of saturated fatty acids (36.61%) in comparison to unsaturated fatty acids (55.69%).

3.3 Physico-chemical Characterization of Fe³⁺-Mn-WO₃/MoO₃

The powder X-ray diffraction has been utilized to examined the crystal structure of the calcined and uncalcined Fe⁺³-Mn-WO₃/MoO₃ catalyst as revealed in Fig. 1a, there are a very small peaks at 20 of (12.1°, 13.6°, 14.9°, 25.7°, 26.8°, 27.6°, 30.4° and 32.9°) (JSPDS file No: 00-052-0561). These diffraction lines revealed lower crystallinity to semi-amorphous FMWMo while, the peaks appeared at 20 = (12.6°, 23.1°, 25.1°, 26.6°, 27.7°, 32.2°, 34.0°, 42.1°, 44.4°, 47.2°, 49.1°, 50.7°, 54.2°, 56.5°, 60.3° and 62.1°) (JSPDS file No: 00-032-1391-Tungsten Molybdenum Oxide) appeared in Fig. 1b indicating that the crystalline structure has a well-established orthorhombic crystal system of FMWMo via interpretation using X’pert high score software. Konya et al. had carried out research in order to relate the performance of Mo₃VO₇ catalyst for the preparation of prechelatom product and they reached to the fact that the orthorhombic Mo₃VO₇ catalyst was revealed the best catalytic activity in the process. The crystallite size of FMWMo had been assessed via Debye-Scherrer relationship and was found to be 33 nm.

Raman spectroscopy is an important tool for the identification of dispersed metal oxides and distinguishes vibrational modes of surface and bulk structures. To analyze the nature of the surface species, laser Raman measurements of ferric-manganese doped tungstated molybdena catalyst calcined at 600°C for 3 h was made. Figure 2 depicts the Raman spectrum result for the calcined FMWMo sample. The assignment of Raman bands was made by comparison with reference standards Kilo et al. Using this informa-

![Fig. 1](image-url)
The Raman band appeared at 434 cm\(^{-1}\) is probably due to the MnO. The Raman spectrum reveals the existence of iron dispersed as Fe\(_2\)O\(_3\). The peaks at 300, 225, 409 and 576 cm\(^{-1}\) match to the literature data for Fe\(_2\)O\(_3\)\(^{19-21}\).

The major vibrational modes of WO\(_3\) were located at 808, 714, and 1112 cm\(^{-1}\) and have been assigned to stretching and bending mode of W-O correspondingly\(^{22}\). Whereas the band observed at 980-1006 cm\(^{-1}\) region was attributed to the terminal Mo=O stretching mode and the bands at 860-910 cm\(^{-1}\) had been assigned to the stretching mode of the Mo-O–Mo bridging bond\(^{23}\).

The overall density of acidic centres as well as a comparative acidity of Fe\(^{3+}\)-Mn-WO\(_3\)/MoO\(_3\) catalyst can be measured by means of a total amount of NH\(_3\) desorbed. The Fe\(^{3+}\)-Mn-WO\(_3\)/MoO\(_3\) catalyst has only one desorption peak at around 308°C which indicates a moderate strength of acidity with about 1117.0 μmol g\(^{-1}\) as a density of the acidic centres (Fig. 3).

Thermogravimetric analysis of uncalcined iron-manganese doped tungstated molybdena as shown in Fig. 4 shows two steps weight loss below 300°C owing to removal of physisorbed, structural water, ammonia, nitrous oxide and complete decomposition of iron and manganese nitrate salts to their oxides (Ferric oxide, Manganese oxide)\(^{24, 25}\) as well as formation of MoO\(_3\) oxide. The third weight losses step at about 830°C is attributed to the evaporation of MoO\(_3\) at higher temperature\(^{25, 26}\).

The specific BET surface area and average size of FMWMo obtained using BET method were 2.1 m\(^2\) g\(^{-1}\) and 84 nm, respectively. In comparison to the average size of triglyceride molecule (5.8 nm), the ferric–manganese doped tungstated molybdena catalyst has a big average pore size (84 nm), the reactants species would be certainly pass through the pores of FMWMo and consequently being more closely to the active sites, thus the best is performance of FMWMo catalyst.

A Shimadzu X-ray fluorescence (XRF) with a Rayny EDX-720 spectrometer was used to find out the fraction of species existing in the catalyst\(^{27, 28}\). The amount of Fe-Mn-doped tungstated molybdena elements were found to be: iron(Fe) –1.542%, manganese (Mn) – 0.521%, tungsten(W) –20.437%, molybdenum (Mo) – 77.5%.

Transmission electron microscopy has been utilized to investigate the dispersion level of FMWMo nanoparticle. The Transmission electron microscopy image of FMWMo is depicted in Fig. 5 which clearly reveals that the FMWMo
nanoparticle have an orthorhombic crystal system with sizes in a limit of 25.8-35.2 nm with average size at 31.8 nm, which matched very well with XRD data. The reproducibility of the method was established through the triplicate preparations and triplicate analyses.

3.4 Optimization of Biodiesel Yield via Fe$^{+3}$-Mn-WO$_3$/MoO$_3$

The influence of different reaction variables containing time, heating effect, quantity of the catalyst in addition to the molar ratio of methanol/waste cooking feedstock on the catalytic activity for simultaneous esterification and transesterification reaction were examined.

3.4.1 Reaction Time

The production of methyl esters versus time was displayed in Fig. 6. The limitation of mass transfer associated with heterogeneous system resulted in slow rate of reaction during commencement of the reaction then the yield increases with time. The optimum duration spent for synthesizing the biodiesel was verified by performing reactions via nanoparticle ferric-manganese doped tungstated molybdena solid acid catalyst at different reaction times. Figure 6 shows the variation of waste cooking oil methyl esters contents with the reaction time. At an optimum considered parameters of the reaction it clears from Fig. 6 that the methyl esters yield was low in the beginning of the reaction until the end of the third hour. Nevertheless, with the progress of time the biodiesel produced had been increased gradually after four hours. However, the reaction approached equilibrium around 92.0 wt% after 8 hours from the start of the reaction. Therefore, 8 h were regarded as an optimum reaction time for synthesis of biodiesel employing spent cooking vegetable oil via nanoparticle ferric-manganese doped tungstated molybdena solid acid catalyst.

3.4.2 Temperature of Reaction

The temperature was found to have drastically controlled the biodiesel yield. Thus, the experiments using the ferric-manganese doped tungstated molybdena nanoparticle solid acid catalyst (FMWMo) were carried out at 80°C up to 220°C with interval of 20°C. The amount of biodiesel formed increased through the increased in the temperature of the procedure, then reached maximum at around 200°C with negligible increased at 220°C as illustrated in Fig. 7. Therefore, the temperature has been held as 200°C for the additional study.

3.4.3 Loading of Catalyst

Catalyst dosing has been recognized as the central reaction factor which wants to investigate deeply so as to improve WCOME's yield. Bigger dosage of the catalyst has been needed to enhance the obtainability of dynamic centres. Nevertheless, effect of FMWMo higher amount (1-7% wt/wt) on the quantity of biodiesel produced has been investigated throughout 8 h, ratio of methyl alcohol/ spent cooking feedstock 25/1, temperature of the process 200°C and speed of mechanical mixer 600 rpm as illustrated in Fig. 8. The rise of catalyst dosage within a limit of 1

Fig. 6 Impact of time progress using 200°C as process temperature, quantity of catalyst 6% ratio of methyl alcohol/ spent cooking feedstock 25/1 and speed of mechanical mixer 600 rpm.

Fig. 7 Impact of temperature of the procedure during 8h, loading 6% wt/wt, ratio of methyl alcohol: waste oil 25:1 and speed of mechanical mixer 600 rpm.

Fig. 8 Impact of Fe(+3)-Mn- WO$_3$/MoO$_3$/ dosage throughout 8h, ratio of methyl alcohol/ spent cooking feedstock 25/1, temperature of the process 200°C and speed of mechanical mixer 600 rpm.
up to 7% wt/wt led to an observable increase in ester’s yield from 65 to 93% ± 0.25. But, once the concentration of catalyst reached 7.0% wt/wt, there was slight increase in biodiesel yield obtained. Thus, it is obvious that the catalyst amount may have a great effect to enhance the yield of biodiesel. Thus 6% wt/wt has been considered as an optimum dosage of the FMWMo.

3.4.4 Impact of Methyl Alcohol/ Spent Cooking Feedstock Molar ratio

Since the rate of simultaneous preparation of spent cooking feedstock based-biodiesel is low, excess amount of methyl alcohol wants to shift the equilibrium forwards. So for the investigation of effect concerning the stoichiometric ratio of methyl alcohol/waste oil feedstock in biodiesel yield, experiments using ferric-manganese doped tungstated molybdena nanoparticle catalyst were carried out by shifting the waste oil feedstock/methyl alcohol stoichiometric ratio in a range of 5–30, while the other parameters have been fixed at their optimum condition. According to the results depicted in Fig. 9, the conversion of waste cooking oil via FMWMo catalyst revealed an obvious reliance on stoichiometric ratio of the reactants. As the amount of the methyl alcohol was enlarged, biodiesel yield had been improved significantly. The highest yield had been gained at stoichiometric ratio of the reactants 25:1; (methyl alcohol/spent oil) further than extra methyl alcohol has irrelevant enhancement on WCOME’s yield.

3.5 The role of Fe\(^{3+}\)-Mn-dopants on the catalytic activity of Fe\(^{3+}\)-Mn\(-\)WO\(_3\)/MoO\(_3\)

The role of ferric and manganese dopants on the surface of ferric-manganese doped tungstated molybdena were determined via the investigation of catalytic activity of the prepared solid acid catalysts (tungstated molybdena and ferric-manganese doped tungstated molybdena (FMWMo) for the preparation of biodiesel simultaneously. The comparative process was carried out at 8 h, stoichiometric ratio of waste oil to methyl alcohol 1:25 with 6% wt/wt. catalyst amount, temperature at 200°C and the 600 rpm stirrer. As a result the experimental yield of methyl esters obtained from tungstated molybdena and ferric-manganese doped tungstated molybdena was found to be 65.0 ± 0.17 and 92.3% ± 1.12, respectively. In addition, Table 1 shows that ferric-manganese doped tungstated molybdena gives highest ester yield as well as specific surface area and density of active centres which indicates that the ferric and manganese dopants increase the surface area, acidity and the catalytic activity of ferric-manganese doped tungstated molybdena relative to that of tungstated molybdena catalyst.

3.6 Reusability of the Catalyst

Catalyst reusability is an input step for commercial feasibility since it decreases the budget of WCOME’s manufacture. Thus, the recycling of the ferric-manganese doped tungstated molybdena nanoparticle catalyst for biodiesel production was studied for several runs after the catalyst had been washed with hexane and then methanol to remove the non-polar and polar compounds and finally dried in an oven at a temperature fixed as maximum as 120°C through six hours. Reusability study was carried out employing the optimum factors of FMWMo catalyst. Figure 10 shows that the product remained almost the same up to the sixth run and then dropped to around 89.1% ± 1.95 in the seventh run suggesting the successful reusability of the catalyst for six runs. The decrease of WCOME’s yield may be assigned to the deactivation of the catalysts due to the partial blockage of the pore by the feedstock and/or the mass lost during the recovery of the catalyst. Therefore, it can be concluded that, the Fe-Mn-doped tungstated 

### Table 1

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Ester yield (%)</th>
<th>BET (m(^2) g(^{-1}))</th>
<th>Density of active sites(µmol g(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>WO(_3)/MoO(_3)</td>
<td>65.0 ± 0.17</td>
<td>1.20</td>
<td>146.46</td>
</tr>
<tr>
<td>Fe-Mn-WO(_3)/MoO(_3)</td>
<td>92.3 ± 1.12</td>
<td>2.10</td>
<td>1117.0</td>
</tr>
</tbody>
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Fig. 9 Impact of the stoichiometric ratio of methyl alcohol/ spent cooking oil, during 8 h, temperature of the methanolyis process 200°C, speed of mechanical mixer 600 rpm and catalyst dosage 6 %w/w.
CONCLUSION

In this study the activity of the Fe-Mn-doped tungstated molybdena nanoparticle catalyst was evaluated for the synthesis of biodiesel from waste cooking oil with high free fatty acid. The WCOME's yield reached 92.3 % ± 1.12 at 8 h reaction, the catalyst amount 6 % wt/wt, and molar ratio of methanol to WCO 25:1 and a temperature of 200 ºC. The catalyst used in this study was found to reduce the high content of FFAs to about 0.620 %, signifying that, the catalyst is highly effective for simultaneous esterification and transesterification reaction. In addition, the recovery and recycling of the Fe-Mn-doped tungstated molybdena in successive catalytic processes indicates that the catalyst can be recycled to six runs without loss in activity. In conclusion, the findings have potential for widespread applications in academic and industrial scale production of biodiesel from waste cooking oil with high FFAs.

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