PREPARATION OF BIODIESEL FROM OIL PALM INDUSTRIAL WASTE USING SULPHATED HETEROGENEOUS CATALYST

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Dedicated to my beloved parents, siblings, supervisors and friends for their never-ending love and encouragement, priceless support and everlasting inspiration towards the success of this study.

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LIST OF ABBREVATIONS

BA	Boiler ash
BET	Brunauer-Emmett-Teller
CPO	Crude palm oil
Dc	Decanter cake
Dc-oil	Decanter cake oil
EFB	Empty fruit bunch
FAME	Fatty acid methyl esters
FESEM	Field emission scanning electron microscope
FFA	Free fatty acids
GC-FID	Gas chromatography-flame ionization detector
GC-MS	Gas chromatography-mass spectrometer
ICP-MS	Inductively coupled plasma mass spectrometer
ME	Methyl esters
MeOH	Methanol
MT	Metric tonnes
PFAD	Palm fatty acid distillate
RBD-PO	Refined, bleached and deodorized palm olein
SBC	Spent bleaching clay
TGA	Thermogravimetry analysis/ Differential thermal analysis
TLC	Thin layer chromatograph
XRD	X-ray diffraction

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ABSTRACT

The use of waste, particularly in the biodiesel industry has gained great potential in countering higher feedstock cost. In the present work, methyl ester was prepared from waste sources, palm fatty acid distillate (PFAD) and decanter cake oil (Dc-oil) via solid acid catalysts (SO_4^{2-}/TiO_2-SiO_2 and $NiSO_4/SiO_2$). In line with the costing related matter, silica from rice husk ash (RHA), an agricultural waste, is used as a support for all the catalysts. The SO_4^{2-}/TiO_2-SiO_2 and $NiSO_4/SiO_2$ catalysts were prepared by wet impregnation method. The obtained results showed that, both prepared catalysts lay in acidic form as tested using Hammett indicators. Both of the catalysts possessed mesoporous structures which were ideal catalyst for biodiesel production. PFAD was successfully esterified using SO_4^{2-}/TiO_2-SiO_2 (calcined at 500 ºC) under the optimal conditions of 2.97 wt.% catalyst amount, 5.85:1 methanol to PFAD molar ratio and reaction time of 3.12 h to produce 93.3% methyl ester. Similarly, 93% methyl ester was achieved using $NiSO_4/SiO_2$ (calcined at 300 ºC) under the optimal conditions of 15 wt.% catalyst amount, 5:1 methanol to PFAD molar ratio and 7 h of methanol refluxing process. As for Dc-oil, SO_4^{2-}/TiO_2-SiO_2 was used to transesterify the oil at optimal conditions of 5 wt.% catalyst amount , 9:1 methanol Dc-oil molar ratio for 5 h response time, producing 60.7% methyl esters. Similarly, 71% methyl esters was achieved using $NiSO_4/SiO_2$ under the optimal conditions of 11 wt.% catalyst amount, 15:1 methanol to oil molar ratio for 9 h reaction time. The prepared SO_4^{2-}/TiO_2-SiO_2 catalyst for the esterification of PFAD was found to be reused up to two times with more than 90% methyl esters produced. On other hand, $NiSO_4/SiO_2$ catalyst for the esterification of PFAD can be reused once only with methyl esters produced about 90%. Besides that, the findings also revealed SO_4^{2-}/TiO_2-SiO_2 had better catalytic activity in esterification of PFAD and transesterification of Dc-oil compared to $NiSO_4/SiO_2$. The investigated solid acid catalysts, SO_4^{2-}/TiO_2-SiO_2 and $NiSO_4/SiO_2$ were found applicable for esterification and transesterification of high FFA feedstock especially from waste feedstock.

ABSTRAK

Penggunaan bahan buangan terutamanaya dalam industri biodiesel adalah berpotensi besar dalam menangani masalah berkaitan kos bahan mentah yang tinggi. Dalam kajian ini, metil ester telah dihasilkan daripda bahan buangan iaitu PFAD dan minyak Dc menggunakan mangkin asid pepejal (SO_4^{2-}/TiO_2-SiO_2 dan $NiSO_4/SiO_2$). Dengan mengambil kira kos pengeluaran biodiesel, silika daripada bahan buangan sektor pertanian iaitu abu sekam padi (RHA) telah dimuatkan ke dalam kedua-dua mangkin yang disediakan. Mangkin SO_4^{2-}/TiO_2-SiO_2 dan $NiSO_4/SiO_2$ telah disediakan melalui kaedah impregnasi basah. Hasil daripada kajian menunjukkan kedua-dua mangkin yang disediakan menunjukkan sifat keasidan apabila diuji dengan indikator Hammett. Selain itu, kedua-dua mangkin mempunyai struktur 'mesopore' yang sangat sesuai sebagai mangkin di dalam penghasilan biodiesel. Mangkin SO_4^{2-}/TiO_2-SiO_2 yang telah dikalsinasi pada suhu 500 ºC telah berjaya mengesterifikasi PFAD dengan peratusan metil ester sebanyak 93.3% pada keadaan tindak balas optimum 5.85:1 nisbah molar metanol kepada PFAD dan berat mangkin sebanyak 2.97% selama 3.12 jam. Keputusan yang sama diperolehi dengan menggunakan mangkin $NiSO_4/SiO_2$ yang dikalsinasi pada suhu 300 ºC, sebanyak 93% metil hasil dapat dihasilkan pada keadaan tindak balas optimum sebanyak 15% berat mangkin dan 5:1 nisbah molar metanol kepada PFAD selama 7 jam masa tindak balas. Mangkin SO_4^{2-}/TiO_2-SiO_2 juga digunakan untuk transesterifikasi minyak Dc dengan metil ester terhasil sebanyak 60.7% pada keadaan optimum berat mangkin sebanyak 5% dan 9:1 nisbah molar metanol kepada minyak Dc selama 5 jam masa tindak balas. Mangkin $NiSO_4/SiO_2$ pula berjaya mengtransesterifikasi minyak Dc kepada metil ester sebanyak 71% pada keadaan optimum iaitu pada berat mangkin sebanyak 11% dan 15:1 nisbah molar metanol kepada minyak Dc selama 9 jam masa tindak balas. Mangkin SO_4^{2-}/TiO_2-SiO_2 yang disediakan terbukti boleh diguna pakai semula sebanyak 2 kali untuk mengesterifikasi PFAD dengan peratusan metil ester terhasil melebihi 90%. Mangkin $NiSO_4/SiO_2$ pula boleh diguna pakai semula sebanyak sekali sahaja dengan metil ester terhasil sebanyak 90%. Selain itu, hasil penemuan kajian ini mununjukkan mangkin SO_4^{2-}/TiO_2-SiO_2 mempunyai aktiviti mangkin yang lebih baik daripada magkin $NiSO_4/SiO_2$ untuk proses esterifikasi PFAD dan transesterifikasi minyak Dc. Hasil kajian juga menunjukkan penggunaan mangkin asid pepejal, SO_4^{2-}/TiO_2-SiO_2 dan $NiSO_4/SiO_2$ boleh diaplikasikan untuk proses esterifikasi dan transesterifikasi bahan mentah yang tinggi kandungan asid lemak (FFA) seperti bahan buangan.

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