TRANSESTERIFICATION OF CRUDE PALM OIL ADSORBED ON SPENT BLEACHING CLAY AND CATFISH OIL USING CaO AND ZnO AS CATALYSTS

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Faculty of Industrial Sciences and Technology
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SEPTEMBER 2016

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LIST OF ABBREVIATIONS

BA	Boiler ash
B-CaO	Calcium oxide form barnacle shell
B-CaO·ZnO	Mixed calcium oxide from barnacle and zinc oxide
BET	Brunauer-Emmett-Teller
B5	5 % blended biodiesel
CaO	Calcium oxide
CPO	Crude palm oil
DC	Decanter cake
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
^{1}H-NMR	Proton nuclear magnetic resonance
ICP-MS	Inductively coupled plasma mass spectrometer
ME	Methyl esters
MeOH	Methanol
O-DC	Oil extracted from decanter cake
PE	Petroleum ether
RBD-PO	Refined, bleached and deodorized palm olein
SBC	Spent bleaching clay
TGA/DTA	Thermogravimetry analysis/ Differential thermal analysis
TLC	Thin layer chromatograph
WPCO	Waste palm cooking oil
XRD	X-ray diffraction
XRF	X-ray fluorescence

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ABSTRACT

In the present work, the transesterification of crude palm oil (CPO) adsorbed on spent bleaching clay (SBC) and waste catfish fat using Commercial-CaO, barnacle (B-CaO) and mixed-oxide (B-CaO·ZnO) as a heterogeneous catalyst were attempted. In order to enhance the catalytic activity, the catalysts have been calcined at 500 - 900 °C for 2 h. It have been had found 900 °C is the optimum condition for catalyst preparation because upon calcination, the catalyst transformed to CaO from the initial $CaCO_3$ structure. The mechanochemical treatment had been used for preparation of mixed oxide. The catalyst had been characterized using thermogravimetric/differential thermal analysis (TGA/DTA), x-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Brunauer, Emmett and Teller surface area (BET), x-ray fluorescence (XRF), field emission scanning electron microscopy (FESEM), energy-dispersive x-ray spectroscopy (EDX) and basic strength using Hammett indicators. Results showed that the CaO exist at the optimum condition of calcination and also found that these catalyst consist of strong basic site. The result shows that, the optimal condition for transesterification of catfish fat and SBC reaction catalyzed by B-CaO at 93.7% and 94.0%, respectively in 4 h reaction duration at 65 °C, methanol to oil molar ratio at 12:1 and 5 wt. % catalysts as an optimal reaction conditions. On the other hand, by using B-CaO·ZnO as catalyst for SBC oil and catfish fat, the optimal conditions were found to be 3 wt.% catalyst, 9:1 methanol to oil molar ratio, yielding 94.7% and 96.3% ME, respectively for 4 h and 2 h of reaction duration at 65 °C. In addition, *in-situ* transesterification using SBC also been carried out where the optimal conditions were found to be 15 wt.% and 25 wt.% of catalyst, 110:1 and 150:1 methanol to oil molar ratio resulting ME content for B-CaO·ZnO and B-CaO, respectively in 6 h and 8 h duration at 65 °C. The catalysts (B-CaO and B-CaO·ZnO) can be reused up to four times maintaining ME content of 65% and 70%, respectively. The methyl esters produced were found to confirm mainly the key specifications of biodiesel. The engine performance of SBC B5 was investigated on a single cylinder 4-stroke diesel engine (YANMAR NF19-SK). The results indicated that the SBC B5 gave lower CO_2 emission compared to neat diesel, thus contributed to the reduction of greenhouse gases. The barnacle shells as a source of calcium oxide can be widely applied as it is or mixed with other oxides as a catalyst in biodiesel production.

ABSTRAK

Transestrifikasi minyak terjerap pada tanah liat yang telah digunakan semasa proses peluntaran SBC dan lemak ikan patin telah dijalankan dengan menggunakan kalsium oksida komersial, kalsium oksida daripada cengkerang teritip (B-CaO) dan gabungannya bersama zink oksida (B-CaO·ZnO) sebagai mangkin. Dalam usaha meningkatkan aktiviti mangkin, mangkin telah dikalsinasi pada suhu 500-900 °C selama 2 jam. Semasa proses kalsinasi, struktur asal $CaCO_3$ telah bertukar kepada CaO. Penyediaan campuran oksida sebagai mangkin telah melalui proses kimia-mekanikal. Karakter mangkin telah dibuktikan dengan menggunakan analisa termogravimetri/ pengkamiran haba, pembelauan x-ray, spektroskopi fourier inframerah, analisa luas permukaan Brunauer, Emmett dan Teller, X-ray pendafluor, pelepasan bidang imbasan mikroskop elektron, tenaga serakan x -ray spektroskop dan kekuatan asas oleh petunjuk Hammet. Peratusan metil ester oleh minyak SBC dan lemak ikan patin dengan menggunakan B-CaO sebagai mangkin adalah 93.7% dan 94.0% selama 4 jam pada suhu 65 °C pada keaadaan tindak balas optimumya ialah 12:1 pada nisbah molar metanol kepada minyak dan 5% mangkin. Sebaliknya, apabila mangkin B-CaO·ZnO digunakan untuk minyak SBC dan lemak ikan patin, keaadaan tindak balas optimum adalah 3% mangkin, 9:1 nisbah molar methanol kepada minyak menghasilkan 94.7% dan 96.3% selama 4 jam untuk minyak SBC dan 4 jam untuk lemak ikan patin pada suhu 65 °C. Selain itu, transesterifikasi *in-situ* dengan menggunakan SBC juga dikaji dimana keadaan tindak balas optimum adalah 15 dan 25 % mangkin, 110:1 dan 150:1 nisbah molar methanol kepada minyak menghasilkan kandungan metil ester 87.3% dan 80.0% untuk mangkin B-CaO dan B-CaO·ZnO selama 6 dan 8 jam pada suhu 65 °C. Kedua-dua mangkin (B-CaO dan B-CaO·ZnO) boleh diguna semula sehingga empat kali sehingga kandungan metil ester adalah 65% (B-CaO) dan 70% (B-CaO·ZnO). Produk metil ester yang dihasilkan menepati beberapa spesifikasi biodiesel. Prestasi enjin untuk campuran 5% biodiesel daripada SBC kepada diesel (SBC B5) dikaji dengan menggunakan enjin diesel bersilinder tunggal 4-stroke (YANMAR NF19-SK). Keputusan menunjukkan SBC B5 membebaskan kandungan karbon dioksida yang rendah berbanding dengan diesel iaitu menyumbang kepada penurunan gas rumah hijau. Cengkerang teritip sebagai sumber utama kalsium oksida sebagai mangkin boleh digunakan secara meluas dalam penghasilan biodiesel secara berasingan ataupun gabungan dengan oksida yang lain.

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