TRANSESTERIFICATION VIA DIFFERENT INTENSIFICATION METHODS USING WASTE FEEDSTOCKS AND WASTE HETEROGENEOUS CATALYSTS

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DOCTOR OF PHILOSOPHY

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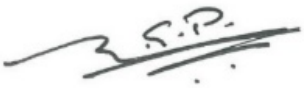
SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and, in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.



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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Kos bahan mentah sangat penting dalam penghasilan biodiesel and ramai pengkaji menjalakan kajian untuk mendapatakan nahan mentah yang lebih murah. Mangkin heterogeny pula membantu dalam penghasilana aor yang minima, memudahakn pemngasingan mangkin dan boleh diguna pakai. Dalam kajian ini, transesterifikasi menggunakan minyak biji Aglaia korthalsii dan minyak ikan patin serta teritip dan logam potassium yang dimuatkan dalam sisa tulang tilapia digunakan sebagai mangkin heterogen. Kalsinasi teritip dan sisa tulang tilapia pada suhu 900 kalsius selama 2 jam telah menukarkan kalsium karbonat kepada kalsium oksida. Kaedah impregnasi basah telah digunakan untuk penyediaan logam potasium yang dimuat pada tulang ikan. Mangkin telah dicirikan dengan termogravimetrik/analisa termal perbandingan (TGA/DTA), X-ray difraksi (XRD), *Fourier transform* spektroskopi inframerah (FTIR), Brunauer, Emmett dan Teller (BET), X-ray *fluorescence*, mikroskop pengimbasan pelepasan medan (FESEM), spektroskopi sinar-X-dispersif tenaga (EDX) dan analisa alkali menggunakan penunjuk Hammett. Keputusan ANOVA menunjukkan kondisi terbaik adalah pada 12:1 nisbah metanol kepada minyak, 4.7 % mengkin selama 3 jam pada suhu 65 celsius. Minyak Aglaia korthalsii berjaya menukarkan minyak keada metil ester sebanyak 97.12 ± 0.49 %. Manakala, lemak ikan berjaya memperoleh sebanyak 93.7 % untuk 3 jam reaksi. Kedua- dua mangkin berjaya digunapakai semula sebanyak 4 kali pada nilai metil ester sebanyak 65 % dan 70 %. Toleransi mangkin terhadap lemk bebas serta kadar kelembapan adalah sebanyak 4 a5 and 3 a5 , respectively. Di antara kaedah penyediaan biodiesel, homogenasi merupakan kaedah terbaik dalam penghasilan kadar tenaga yang paling rendah.Prestasi enjin untuk campuran 5% biodiesel daripada minyak masak terbuang yang dimangkin oleh K-FB900 dikaji dengan menggunakan enjin diesel bersilinder tunggal 4-stroke.Kajian telah membuktikan Biodeiesel membantu dalam menurunkan kadar haba dan penggunaan tenaga yang lebih rendah.

ABSTRACT

Feedstock cost is one of the main attentions in biodiesel production and researchers intensively seeking for a low-cost feedstock in order to make the process viable. Heterogenous catalyst helps offer less water generation, ease the catalyst separation and able to reused. In this present work, the transesterification of *Aglaia korthalsii* seed oil and catfish fat using barnacle and potassium loaded on tilapia fish bone as heterogeneous catalysts were attempted. It has been found that 900 °C at 2 h calcination is the optimum condition for barnacles and tilapia fish bone to transform the $CaCO_3$ into CaO. While, 10% potassium had successfully loaded on tilapia fish bone using wet impregnated treatment. 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. ANOVA results revealed that the experimental model is well significant and under the suggested optimal reaction conditions of 12.2:1 MeOH:oil molar ratio and 4.7 wt.% catalyst (3 h reaction duration at 65 °C), *Aglaia korthalsii* seed oil was successfully converted into methyl ester with highest conversion of 97.12 ± 0.49 wt.%. Whereas the catfish fat shows the conversions at 93.7 wt.% for 3 h reaction duration at 65 °C, MeOH: oil molar ratio at 12:1 and 5 wt.% catalyst as an optimal reaction condition. Both catalysts can be reused up to four times maintaining methyl ester content of 65% and 70%, respectively. The catalyst could tolerate up to maximum level of 4% free fatty acid and 3% moisture with the methyl ester conversion above 94 wt.%. Among all methods, homogenizer show the best activation energy below 30 kJ/mol. The methyl esters produced were found to conform the key specifications for biodiesel follow the EN 14103 standard. The engine performance of blended 5 % biodiesel was investigated on a single cylinder 4-stroke diesel engine. The results indicated that the blended 5 % biodiesel gave maximum fuel consumption up to 1.7 kg/h whereas the neat biodiesel was at 1.45 kg/h. It has been proven that biodiesel will help in reducing the heating value and lower the energy input.

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