



UNIVERSITI PUTRA MALAYSIA

**APPLICATION OF TAGUCHI METHOD IN THE OPTIMIZATION OF
BIODIESEL PRODUCTION FROM WASTE COOKING OIL USING
MoO₃/SiO₂ CATALYSTS**

NUR SHARINA BINTI ANWAR

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment
of the requirement for the degree of Master of Science

**APPLICATION OF TAGUCHI METHOD IN THE OPTIMIZATION OF
BIODIESEL PRODUCTION FROM WASTE COOKING OIL USING
MoO₃/SiO₂ CATALYSTS**

By

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May 2012

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Today, edible oil or cooking oil is one of the most important elements in food production industry as well as food preparation at home. As a consequence, large amount of waste cooking oil (WCO) was produced especially in developing countries. With increasing fossil fuel price and decreasing renewable energy supply, it is of main interest to utilize the WCO by converting it into usable and beneficial product which is biodiesel. Heterogeneous acid catalyst which can simultaneously carry out esterification and transesterification reaction was selected for converting that kind of low grade oil into biodiesel. In this study, sulfated MoO₃/SiO₂ with different loading percentage of MoO₃ were synthesized via impregnation method and applied in the transesterification reaction of WCO to biodiesel. X-ray Diffraction (XRD), Brunauer-Emmett-Teller (BET) surface area and Ammonia-Temperature Programme Desorption (NH₃-TPD) were used to characterize the catalysts synthesized while the biodiesel produced were analyzed by proton Nuclear Magnetic

Resonance (^1H NMR) and Gas Chromatography-Flame Ionization Detector (GC-FID). The transesterification reaction was optimized by Taguchi method in which L_{16} orthogonal array involving four levels and five parameters was employed to investigate the effect of each reaction parameters which are reaction temperature, reaction time, methanol to WCO molar ratio, catalyst amount and MoO_3 loading on SiO_2 and simultaneously determine the optimum conditions for the transesterification reaction. Results showed that the reaction temperature, reaction time, methanol to WCO molar ratio and catalyst amount were the significant influential parameters that influence the final yield of biodiesel while MoO_3 loading on SiO_2 is insignificant parameter. The optimum conditions of the reaction were derived to be at 453 K reaction temperature, 6 h reaction time, 30:1 methanol to WCO molar ratio, 1 wt% of the catalyst amount and 3 wt% MoO_3 loading on the SiO_2 with 98.56 % biodiesel yield obtained which matched well with predicted yield. Some of physicochemical properties of the biodiesel produced were analyzed and the results were complied with the international standard specification requirements. Another study on the catalyst performance shows that the catalyst exhibits good stability and reusability up to at least five times in transesterification reaction without significant lost in activity. The overall results indicate the biodiesel produced exhibits good properties and suitability to be produced industrially.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**APLIKASI KAEDAH TAGUCHI DALAM MENGOPTIMAKAN
PENGHASILAN BIODIESEL DARIPADA MINYAK MASAK TERPAKAI
MENGUNAKAN MANGKIN $\text{MoO}_3/\text{SiO}_2$**

Oleh

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Hari ini, minyak yang boleh dimakan atau minyak masak merupakan salah satu dari elemen penting dalam industri pembuatan dan penyediaan makanan di industry dan juga di rumah. Akibatnya, sejumlah besar minyak masak terpakai telah dihasilkan terutamanya di negara-negara membangun. Dengan kenaikan harga petrol dan penurunan bekalan tenaga yang boleh diperbaharui, ia menjadi tarikan utama untuk menggunakan sisa tersebut dengan menukarkannya menjadi satu produk yang berguna dan berfaedah iaitu biodiesel. Mangkin asid heterogen yang mampu menjalankan tindakbalas pengtransesteran dan transesteran sekali gus pada masa yang sama telah dipilih untuk digunakan dalam penukaran minyak berkualiti rendah tersebut kepada biodiesel. Dalam penyelidikan ini, $\text{MoO}_3/\text{SiO}_2$ tersulfat dengan peratusan sebatian MoO_3 termuat yang berbeza telah dihasilkan melalui kaedah pengisitepuan dan diaplikasikan dalam tindakbalas pengtransesteran minyak masak terpakai kepada biodiesel. Pembelauan sinar-X (XRD), pengukuran luas permukaan Brunauer-Emmett-Teller (BET) dan penyahjerapan amonia pada suhu terkawal

(NH₃-TPD) telah digunakan untuk mencirikan mangkin yang telah terhasil manakala biodiesel yang disediakan pula dicirikan dengan menggunakan resonan magnetik nuklear proton (¹H NMR) dan kromatografi gas dilengkapi pengesanan pengionan nyala (GC-FID). Keadaan pemboleh ubah tindakbalas pengtransesteran telah dioptimumkan dengan menggunakan kaedah Taguchi dimana susunan ortogonal L₁₆ yang melibatkan empat peringkat dan lima pemboleh ubah telah digunapakai untuk mengkaji kesan setiap pemboleh ubah termasuk suhu tindakbalas, masa tindakbalas, nisbah molar metanol terhadap minyak masak terpakai, kuantiti mangkin dan peratusan MoO₃ dimuat ke dalam SiO₂ sekali gus menentukan keadaan optimum untuk tindakbalas pengtransesteran tersebut. Keputusan menunjukkan suhu tindakbalas, masa tindakbalas, nisbah molar metanol terhadap minyak masak terpakai serta kuantiti mangkin merupakan pemboleh ubah berpengaruh yang ketara yang telah mempengaruhi hasil biodiesel manakala peratusan MoO₃ dimuat ke dalam SiO₂ merupakan pemboleh ubah yang tidak ketara. Keadaan optimum dalam tindakbalas tersebut telah ditentukan pada suhu tindakbalas 453 K, 6 jam masa tindakbalas, 30:1 nisbah molar metanol terhadap minyak masak terpakai, 1 % kuantiti mangkin dan 3 % MoO₃ termuat dalam silica dengan penghasilan biodiesel sebanyak 98.56 % yang juga berpadanan tinggi dengan hasil jangkaan. Beberapa sifat fisikokimia biodiesel yang disediakan juga telah dianalisis dan keputusannya mematuhi keperluan piawaian antarabangsa. Satu lagi kajian terhadap persembahan mangkin menunjukkan mangkin tersebut mempunyai kestabilan yang baik dan boleh digunakan semula sekurang-kurangnya sehingga lima kali dalam tindakbalas pengtransesteran tanpa kejatuhan yang ketara dalam aktivitiya. Keputusan keseluruhan menunjukkan biodiesel yang disediakan mempunyai sifat yang baik dan bersesuaian untuk dihasilkan secara industri.

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I certify that a Thesis Examination Committee has met on 22 May 2012 to conduct the final examination of Nur Sharina binti Anwar on her thesis entitled "Application of Taguchi Method in the Optimization of Biodiesel Production from Waste Cooking Oil using MoO₃/SiO₂ Catalysts" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science – Catalysis.

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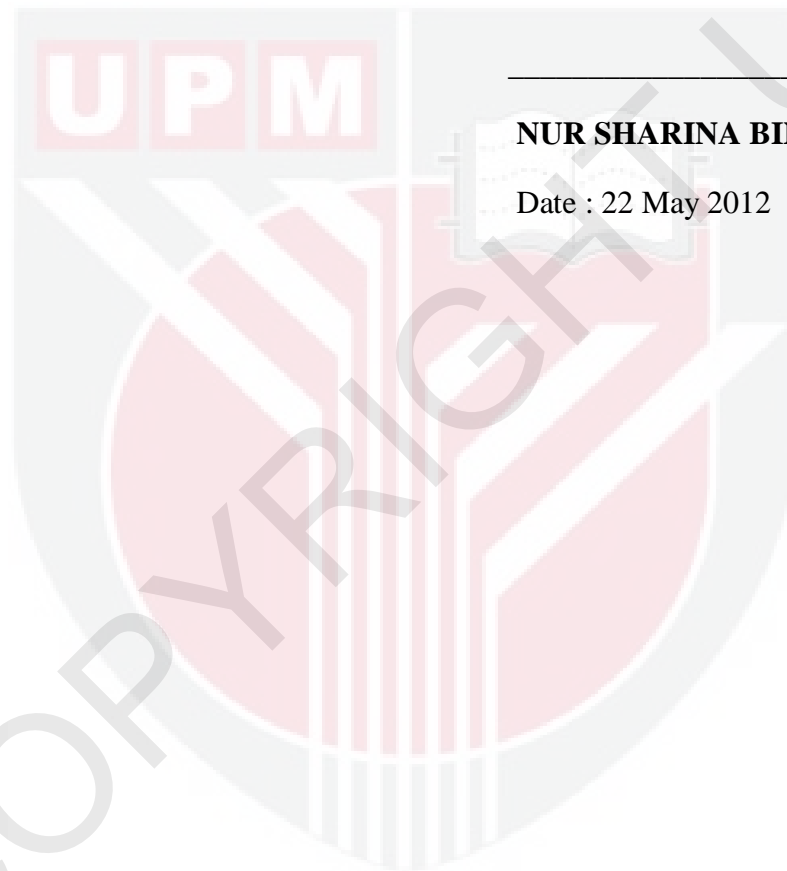
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DECLARATION

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



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