

SYNTHESIS OF CATALYST FROM  
EGGSHELL AND PERNA V. SHELL FOR  
BIOLUBRICANT PRODUCTION

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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 Master of Science.

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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

Pada masa ini, isu-isu pemuliharaan alam sekitar telah meningkat disebabkan oleh penggunaan minyak pelincir yang berasaskan minyak sintetik dan mineral secara meluas. Penggunaan secara terus minyak sayuran sebagai minyak asas minyak pelincir untuk mengelakkan isu-isu alam sekitar tidak efisien kerana kestabilan pengoksidaan, haba dan hidrolitik minyak sayuran yang lemah. Transesterifikasi digunakan secara meluas di industri untuk menangani batasan ini. Dalam usaha untuk menangani masalah yang timbul dengan penggunaan pemangkin homogen, kajian kini tertumpu pemangkin heterogen iaitu kalsium oksida daripada sisa kulit telur dan *Perna V*. Prestasi pemangkin ini telah diuji. Pemangkin daripada kulit telur dan *Perna V* telah disediakan dan diuji untuk mengenalpasti suhu penguraian, morfologi permukaan, luas permukaan dan corak pembelauan X-Ray dan komposisi unsur pemangkin. Dua langkah tindak balas transesterifikasi telah dikaji untuk menukar minyak jarak ke dalam produk akhir iaitu minyak jarak trimethylolpropane triester. Produk perantaraan tertinggi iaitu minyak jarak methyl ester yang tertinggi diperolehi pada masa reaksi 2 jam, metanol kepada nisbah minyak 6: 1, suhu operasi 65° dan 2wt /% berat pemangkin. Pemangkin berkesan untuk tindak balas transesterifikasi pemangkin pertama adalah kalium yang dihamilkan *Perna V*. Pemangkin kulit *Perna V* bersama kalium memberi hasil 92.2%. Manakala bagi keadaan operasi optimum bagi tindak balas transesterification pemangkin kedua antara minyak kastor methyl ester dan trimethylolpropane berada di 120°C, masa tindak balas 3 jam dengan 3wt / wt% daripada kepekatan pemangkin dan 4: 1 TMP untuk nisbah molar. Pemangkin terbaik untuk tindak balas transesterification kedua adalah kalium berubat telur pemangkin dengan 72.4% daripada komposisi triester. Bagi kedua-dua tindak balas transesterifikasi, pemangking cengkerang yang telah dihamilkan adalah lebih baik daripada cengkerang yang tidak dihamilkan kerana terdapat banyak tempat aktif untuk mempercepatkan reaksi. Ester trimethylolpropane yang dihasilkan sejajar dengan piawaian minyak pelincir untuk kegunaan hydrolic.

## ABSTRACT

Currently, environment conservation issues had been rising due to the extensive usage of mineral and synthetic oil based lubricants. The direct application of vegetable oil as biolubricant base oil to overcome these environmental concerns had some drawbacks due to poor oxidation, thermal and hydrolytic stability of the vegetable oil. Catalytic transesterification is widely used in industry to tackle these limitations. In order to tackle the problems arise with the usage of existing homogeneous and heterogeneous catalyst, this study focused on employment of non- and potassium hydroxide impregnated heterogeneous calcium oxide catalyst from waste egg and *Perna V.* shell catalysts. The potential of impregnated waste based catalyst in the synthesis of biolubricant still have not been explored. The performances of these catalysts were being tested. The non- and impregnated egg and *Perna V.* catalysts were prepared and the decomposition temperature, surface morphology, surface area and X-Ray diffraction patterns and elemental composition of the catalysts were investigated. The two steps of transesterification reaction of castor oil into final product of castor oil trimethylolpropane triester were researched. The production of biolubricant consists of two catalytic transesterification reactions in series with castor oil methyl ester as intermediate product. The highest intermediate product, castor oil methyl ester were obtained at reaction time of 2 hours, methanol to oil ratio of 6:1, operation temperature of 65° and 2wt/wt% of catalyst loading. The effective catalyst for this reaction was potassium impregnated *Perna V.* shell catalyst with castor oil methyl ester yield of 92.2%. The optimum operation conditions for second catalytic transesterification reaction between castor oil methyl ester and trimethylolpropane was at 120°C, reaction time of 3 hours with 3wt/wt% of catalyst concentration and 4:1trimethylolpropane to castor oil methyl ester molar ratio. The best catalyst for the second transesterification reaction was potassium impregnated eggshell catalyst with 71.4% of triester composition. For both the reaction, the impregnated catalyst performed better than the non-impregnated catalysts due to the presence of enhanced active sites for the transesterification to take place. The produced trimethylolpropane esters followed the biolubricant standards for hydraulic purposes.

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## LIST OF SYMBOLS

°C	Degree Celcius
h	Hour
min	Minute
%	Percentage

## LIST OF ABBREVIATIONS

BDL	Below Detectable Level
BET	Brunauer-Emmet-Teller
COME	Castor oil methyl ester
CaO	Calcium oxide
COTT	Castor oil trimethylolpropane triester
CCD	Central composite design
FFA	Free fatty acid
FTIR	Fourier transform infra-red spectrometry
GC-FID	Gas chromatography – flame ionization detector
GC-MS	Gas chromatography - mass spectrometry
ICEPS	Impregnated chicken egg and <i>Perna V.</i> shell catalysts
IES	Impregnated egg shell catalyst
IPS	Impregnated <i>Perna V.</i> shell catalyst
ISO VG	International Standard Organization Viscosity Grade
KOH	Potassium hydroxide
SEM	Scanning electron microscopy
TGA-DTA	Thermogravimetric and differential thermal analysis
TMP	Trimethylolpropane
XRD	X-Ray diffraction
XRF	X-Ray fluorescence

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