UNIVERSITI PUTRA MALAYSIA

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COMPARISON OF PALM OIL ESTERS PRODUCTION USING IMMOBILIZED LIPASES, RM 1M AND TL 1M

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Master of Science

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COMPARISON OF PALM OIL ESTERS PRODUCTION USING IMMOBILIZED LIPASES, RM 1M AND TL 1M

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June 2012

Chairman : Professor Mahiran Basri, PhD

Faculty : Science

Palm oil esters (POE) were synthesized via enzymatic alcoholysis of palm oil fractions with oleyl alcohol using commercialized immobilized lipase as biocatalysts. The research focused on attempting to produce POE in a larger scale and reducing the production cost by replacing Lipozyme RM IM with Lipozyme TL IM which is 6 times cheaper as the alternative biocatalyst. The reaction condition used was based on the optimized condition of Lipozyme RM IM. Optimization was conducted in 2-L Stirred Tank Reactor (STR) using one parameter at a time approach. The optimum alcoholysis condition for Lipozyme TL IM in 2-L STR was: amount of palm oil 600 mmol, amount of oleyl alcohol 1800 mmol, amount of enzyme 24.7wt% of palm oil, temperature 60°C, reaction time 3 h and impeller speed 275 rpm.

Using the optimum condition for production of POE using Lipozyme RM IM and Lipozyme TL IM, the reaction was scaled-up from lab scale (2-L STR) to higher scale production (15-L, 75-L and 300-L STR). For scale up using Lipozyme RM IM, the

production was successfully scaled-up from 2-L to 15-L STR with similar result, But with production size of 300-L STR, the yield was reduced to 10%. Reduction of yield was also observed when Lipozyme TL IM was used. The yield decreased 13% during up scaling from 2-L to 15-L STR and another 30% from 15-L to 75-L STR.

The transfer of downstream processing of the POE produced from the conventional separating funnel to stirred tank reactor to perform liquid-liquid extraction process was also investigated. The extraction condition was adopted from previous study. Effect of the impeller speed was studied and showed that there was no significant increment in percentage of extraction of oleyl alcohol with increase of impeller speed from 150-300 rpm. Effect of extraction cycle showed that palm oil esters were purified completely with 4 extraction steps. In the first extraction step, 65% of oleyl alcohol was successfully extracted.

A kinetic study of the alcoholysis reaction of palm oil and oleyl alcohol using Lipozyme TL IM was conducted in a 2-L STR with the proposed mechanism of Ping-Pong Bi-Bi. Within the substrate concentration studied, the reaction obeyed the proposed mechanism without inhibition from the substrate (V_{max} = 32.7 mmol/min, $K_{m(Palm\ Oil)}$ = 0.3147 mmol/ml, $K_{m(Oleyl\ Alcohol)}$ = 0.9483 mmol/ml). Thermodynamic parameters were as follows: ΔH^{0} = 16 803.402 J/mol, ΔS^{0} = 72.039 $J.mol^{-1}.K^{-1,}$ Activation Energy, E_a = 16 850.396 J/mol.

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

PERBANDINGAN PENGHASILAN ESTER MINYAK SAWIT MENGGUNAKAN LIPASE TERSEKATGERAK, RM 1M DAN TL 1M

Oleh

MOHD AZLAN BIN KASSIM

Jun 2012

Pengerusi : Profesor Mahiran Basri, PhD

Fakulti : Sains

Ester minyak sawit (POE) telah disintesis melalui tindakbalas alkoholisis menggunakan lipase komersil tersekatgerak dengan minyak kelapa sawit dan oleil alkohol. Kajian yang dijalankan memfokus kepada penghasilan POE pada skala yang lebih besar dan mengurangkan kos penghasilan POE dengan menggantikan penggunaan Lipozyme RM IM kepada Lipozyme TL IM yang 6 kali lebih murah. Pengoptimuman keadaan tindak balas menggunakan Lipozyme TL IM dijalankan di dalam reaktor tangki berpengaduk 2-L menggunakan pendekatan satu parameter pada satu masa. Keadaan tindak balas alkoholisis optima bagi penggunaan Lipozyme TL IM adalah seperti berikut: jumlah minyak kelapa sawit 600 mmol, jumlah oleil alkohol 1800 mmol, jumlah lipase tersekatgerak 24.7% berat minyak kelapa sawit, suhu tindak balas 60°C, tempoh tindak balas 3 h, kelajuan pengaduk 275 rpm.

Menggunakan keadaan tindak balas optimum bagi pengeluaran POE menggunakan Lipozyme RM IM dan Lipozyme TL IM, tindak balas telah di skala naik daripada skala makmal (2-L STR) kepada skala pengeluaran yang lebih besar (15-L, 75-L dan 300-L STR). Bagi pengskalaan ke atas tindak balas menggunakan Lipozyme RM IM, proses pengeluaran berjaya diskala naik dari 2-L ke 15-L STR dengan kadar hasil yang hampir sama. Bagi penghasilan pada skala 300-L STR menunjukkan penyusutan kadar hasil sebanyak 10%. Penyusutan kadar hasil turut diperhatikan pada proses pengskalaan naik tindak balas menggunakan Lipozyme TL IM. Penyusutan sebanyak 13% diperhatikan pada skala 2-L ke 15-L STR dan 30% pada skala 15-L ke 75-L STR.

Pemindahan kepada proses hiliran telah dikaji daripada menggunakan corong pemisah kepada penggunaan reaktor tangki berpengaduk. Keadaan pengekstrakan adalah berdasarkan kajian terdahulu. Kesan kelajuan pengaduk kepada kadar hasil pengektrakan oleil alkohol telah dikaji dan menunjukan tiada peningkatan yang ketara terhadap kadar hasil pengekstrakan oleil alkohol dengan peningkatan kelajuan pengaduk dari 150 ke 300 rpm. Kesan bilangan kitaran pengekstrakan menunjukkan oleil alcohol berjaya diekstrak sepenuhnya dalam 4 kitaran.

Kajian kinetik bagi tindak balas alkoholisis antara minyak kelapa sawit dan oliel alkohol bermangkin oleh Lipozyme TL IM telah dijalankan dan mencadangkan Ping Pong Bi Bi sebagai mekanisma tindak balas. Di dalam julat kepekatan substrat tindak balas yang dikaji, keputusan menunjukkan bahawa tindak balas alkoholisis minyak kelapa sawit dan oleil alkohol mematuhi mekanisma yang dicadangkan tanpa sebarang perencatan. (V_{maks} = 32.7 mmol/min, $K_{m(Minyak\ Kelapa\ Sawit)}$ = 0.3147 mmol/ml, $K_{m(Oleil\ Alkohol)}$ = 0.9483

mmol/ml). Parameter termodinamik adalah seperti berikut:: $\Delta H^{0} =$ 16 803.402 J/mol, $\Delta S^{0} =$ 72.039 J.mol^{-1}.K$^{-1,}$ Tenaga Pengaktifan, E_a = 16 850.396 J/mol.

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I certify that a Thesis Examination Committee has met on 11 June 2012 to conduct the final examination of Mohd Azlan Bin Kassim on his Master of Science thesis entitled "Comparison of Palm Oil Esters Production Using Immobilized Lipases, RM IM and TL IM" in accordance with the Universities and University College Act 1971 and the Constitution of the Universiti Pertanian Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the degree of Master of Science, MSc.

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DECLARATION

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

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MOHD AZLAN BIN KASSIM

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