UNIVERSITI PUTRA MALAYSIA

CATALYTIC CONVERSION OF GLYCEROL TO VALUE-ADDED ACETOL AND PROPYLENE GLYCOL

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By

MOHD HANIF MOHAMAD

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

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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

CATALYTIC CONVERSION OF GLYCEROL TO VALUE-ADDED ACETOL AND PROPYLENE GLYCOL

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June 2013

Chairman: **Nor Azowa binti Ibrahim, PhD**

Faculty: **Science**

Glycerol conversion to value-added product, acetol was studied through dehydration reaction of glycerol catalyzed by copper chromite in a single step reactive distillation. The physical properties of the synthesized acetol such as its appearance, density and viscosity were compared with those of a commercial product. The effects of reaction temperature, reaction period, catalyst loading and water content in glycerol feedstock were studied to optimize its production. The results show conversion of glycerol increased with the increase of reaction period and catalyst loading while the presence of water in glycerol feedstock decreases the conversion and also reduces the residue which provides ease removal and recycles the catalyst. Optimum conditions for the conversion of glycerol to acetol was at reaction period of 3.5 hours with 2.5% catalyst concentration and 200°C reaction temperature without dilution where the 64.47% product was obtained.

Fourier-Transform infrared (FTIR), gas chromatography (GC), gas chromatography-mass spectroscopy (GC/MS) and nuclear magnetic resonance (NMR) were used to

characterize and validate the product from the dehydration reaction of glycerol. The IR spectrum of product showed stretching of carbonyl group suggested that acetol has been successfully synthesized. The GC-MS analysis showed the value expected for the molecular ion of isolated acetol. The NMR was also used to validate the number and position of carbon and hydrogen in the product by evaluates the chemical shifts.

In brief, the synthesis of propylene glycol from acetol and glycerol has successfully been carried out through hydrogenation reaction catalyzed by copper chromite catalyst. Acetol produces the highest conversion to propylene glycol with 88.43% conversion and 65.22% product yield in 24 hours reaction period, 5% catalyst concentration and 200 psi hydrogen pressure at 200°C reaction temperature respectively. Meanwhile, the propylene glycol produces from glycerol yield 61.80% product with the conversion percentage of 72.77% at the same reaction conditions.

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

PENUKARAN BERMANGKIN GLISEROL KEPADA PRODUK CECAIR ASETOL DAN PROPILENA GLIKOL

Oleh

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Penukaran gliserol kepada produk bernilai tinggi, asetol telah dikaji melalui tindak balas nyah-hidrat gliserol dimangkin oleh kuprum kromit dalam penyulingan reaktif satu langkah. Ciri-ciri fizikal asetol yang dihasilkan seperti penampilan, ketumpatan dan kelikatan telah dibandingkan dengan produk komersil. Kesan suhu tindak balas, tempoh tindak balas, kuantiti mangkin dan kandungan air dalam stok suapan gliserol dikaji untuk mengoptimumkan penghasilannya. Hasil kajian menunjukkan penukaran gliserol meningkat dengan peningkatan tempoh tindak balas dan kuantiti mangkin manakala kehadiran air dalam stok suapan gliserol merendahkan penukaran dan juga mengurangkan sisa dimana memudahkan pengasingan dan kitar semula mangkin. Keadaan optimum untuk penukaran gliserol kepada asetol adalah pada tempoh tindak balas sebanyak 3.5 jam dengan kuantiti mangkin 2.5% dan 200°C suhu tindak balas tanpa pencairan stok suapan di mana ia memperolehi 64.47% produk.

Inframerah Transformasi Fourier (FTIR), kromatografi gas (GC), kromatografi gas-spektroskopi jisim (GC/MS) dan resonans magnetik nuklear (NMR) telah digunakan untuk mencirikan dan mengesahkan produk daripada tindak balas nyah-hidrat gliserol. Spektrum IR produk menunjukkan regangan kumpulan karbonil menunjukkan bahawa asetol telah berjaya dihasilkan. Analisis GC/MS menunjukkan nilai seperti yang dijangka bagi ion terpencil molekul asetol. NMR juga digunakan untuk mengesahkan bilangan serta kedudukan karbon dan hidrogen dalam produk melalui anjakan kimia.

Secara ringkas, sintesis propilena glikol dari asetol dan gliserol telah berjaya dilakukan melalui tindak balas penghidrogenan yang dimangkinkan oleh kuprum kromit. Asetol menghasilkan peratusan penukaran kepada propilena glikol dengan 88.43% dan menghasilkan 65.22% produk dalam 24 jam tempoh tindak balas, 5% kuantiti pemangkin, 200 psi tekanan hidrogen pada 200°C suhu tindak balas. Sementara itu, propilena glikol yang dihasilkan daripada gliserol menghasilkan 61.80% produk dengan 72.77% peratusan penukaran pada keadaan tindak balas yang sama.

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I certify that a Thesis Examination Committee has met on 28th June 2013 to conduct the final examination of Mohd Hanif bin Mohamad on his thesis entitled "Catalytic Conversion of Glycerol to Value-added Acetol and Propylene Glycol" 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.

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The thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement of the degree of Master of Science. The members of supervisory committee were as follows:

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DECLARATION

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

MOHD HANIF BIN MOHAMAD

Date: 28 June 2013

TABLE OF CONTENTS