ACETYLATION OF GLYCEROL OVER CARBON SUPPORTED YTTRIUM OXIDE CATALYSTS AND OPTIMIZATION ANALYSIS BY RESPONSE SURFACE METHODOLOGY

By

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The use of homogeneous catalysts in glycerol acetylation have been associated with several shortcomings hence the growing interest in the use of heterogeneous catalysts such as metal oxide. Carbon supported yttrium oxide (Y_2O_3) catalysts were synthesized via carbonization of palm kernel shell (PKS) and subsequent functionalization with 5wt%, 10wt% and 15wt% yttrium oxide (Y_2O_3), respectively. The catalysts were also characterized by N_2 physisorption analysis (BET surface area), X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX), Fourier transform infra-red (FTIR), and temperature programmed desorption-ammonia (TPD-NH_3) and tested in glycerol acetylation. The 15wt% Y_2O_3/PKS-T700 catalyst with the highest potential was used for the optimization reaction using four-factor, two-level face-centred central composite design (2^4 CCD) of the response surface methodology. The optimized conditions were found to be temperature 130 ℃, glycerol-to-acetic acid molar ratio 1:11 and catalyst loading 0.5 g in 5 h reaction time. Glycerol acetylation reaction revealed optimal results of 99.8% glycerol conversion (GC) and product selectivity of 15.7% monoacetin (MA), 58.8% diacetin (DA), and 29.4% triacetin (TA), respectively. The model terms were found to be significant ($p<0.05$) with coefficient of determination (R^2) close to unity (>0.99) while the predicted R^2 and the adjusted R^2 were in agreement with each other. The characteristics of the catalyst revealed carbon, hydrogen and yttrium were 71.33%, 15.66% and 13.01%, acid site density of 339.9 µmol/g, while the BET surface area, pore volume and the average pore size were 503 m^2/g, 0.452 cm^3/g and 2.5 nm. On subjecting the catalyst to reusability study in three (3) reaction cycles under the optimal conditions, the catalyst was found to maintain good reaction with little degradation as observed in the small decline in performance attributed to slight leaching of the active site.

Abstrak tesis yang dikemukakan kepada Senat of Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

PENGASETILAN GLISEROL KE ATAS MANGKIN KARBON TERSOKONG ITRIUM OKSIDA DAN ANALISIS PENGOPTIMUMAN MENGGUNAKAN KAEDAH GERAK BALAS PERMUKAAN

Oleh

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Penggunaan mangkin homogen dalam asetilasi gliserol telah dikaitkan dengan beberapa kelemahan, justeru minat yang semakin meningkat dalam penggunaan mangkin heterogen seperti oksida logam. Pemangkin itrium oksida (Y_2O_3) yang disokong karbon telah disintesis melalui pengkarbonan tempurung isirong sawit (PKS) di ikuti dengan kefungsian masing-masing 5 bt%, 10 bt% dan 15 bt% itrium oksida (Y_2O_3). Pemangkin juga dicirikan oleh analisis fizijerapan N_2 (luas permukan BET), pembelauan sinar-X (XRD), analisis termogravimetrik (TGA), mikroskop elektron pengimbasan ditambah dengan spektroskopi sinar-X penyebaran tenaga (SEM-EDX), infra-merah transformasi Fourier (FTIR), dan suhu terprogram nyahjerapan-ammonia (TPD-NH_3) dan diuji dalam pengasetilan gliserol. Pemangkin 15bt% Y_2O_3/PKS-T700 dengan potensi tertinggi digunakan untuk tindak balas pengoptimuman menggunakan reka bentuk komposit berpusat dua peringkat (2^4 CCD) bagi kaedah gerak balas permukaan empat faktor. Keadaan optimum di dapati ialah suhu 130 ℃, nisbah molar gliserol/asid asetik 1:11 dan muatan mangkin 0.5 g dalam masa tindak balas 5 jam. Tindak balas pengasetilan gliserol menunjukkan keputusan optimum 99.8% penukaran gliserol (GC) dan selektiviti produk masing-masing 15.7% monoasetin (MA), 58.8% diasetin (DA), dan 29.4% triasetin (TA). Terma model di dapati signifikan ($p<0.05$) dengan pekali penentuan (R^2) menghampiri perpaduan (>0.99) manakala R^2 yang diramalkan dan R^2 terlaras adalah selaras antara satu sama lain. Ciri-ciri pemangkin mendedahkan karbon, hidrogen dan itrium ialah 71.33%, 15.66% dan 13.01%, ketumpatan tapak asid 339.9 μmol/g, manakala luas permukaan BET, isipadu liang dan saiz liang purata ialah 503 m^2/g, 0.452 cm^3/g dan 2.5 nm. Semasa menyasarkan mangkin kepada kajian kebolehgunaan semula dalam tiga (3) kitaran tindak balas di bawah keadaan optimum, mangkin didapati mengekalkan tindak balas yang baik dengan sedikit degradasi seperti yang diperhatikan dalam penurunan kecil dalam prestasi disebabkan oleh sedikit larut lesap tapak aktif.

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

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LIST OF SYMBOLS/UNITS

α	Alpha
β	Beta
℃	Degree Celsius
%	Percentage
µL	Microlitre
µm	Micrometre
µmoles	Micromoles
µA	Microampere
g	Gram
ppm	Parts per million
L	Litre
M	Molar
m^2/g	Square metre per gram
cm^3/g	Cubic centimetre per gram
nm	nanometre
mmol/g	millimoles per gram
g/cm^3	Gram per cubic centimetre

LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
BET	Brunauer-Emmett-Teller
BJH	Barrett–Joyner–Halenda
CCD	Central composite design
DA	Diacetin
DI	Deionized water
DOE	Design of experiment
EDX	Energy dispersive X-ray spectroscopy
FAME	Fatty acid methyl ester
FD	Factorial design
FFA	Free fatty acid
FESEM	Field emission scanning electron microscope
FTIR	Fourier transform infrared spectroscopy
G/AA	Glycerol-to-acetic acid molar ratio
GC	Glycerol conversion
GC-FID	Gas chromatograph-Flame ionization detector
GC-MS	Hydrothermal carbonization
HC	Internal diameter
ID	International union of pure and applied chemistry
IUPAC	Monoacetin
MA	One factor at a time
OFAT	Palm kernel shell
PKS	Scanning electron microscope
SEM	Response surface methodology
RSM	Triacetin
TA	Thermal conductivity detector
TCD	Thermogravimetric analysis
TGA	Temperature programme desorption of ammonia
TPD-NH_3	X-ray diffraction
XRD	

CHAPTER 1

INTRODUCTION

1.1 Research background

Glycerol (propane-1,2,3-triol) is a sweet tasting oily liquid with three hydrophilic hydroxyl groups each attached to carbon responsible for its stable, versatile reactions and applications (Ayoub and Abdullah, 2012). Prior to the advent of biodiesel production, glycerol has a relatively good market value due to the fact that their production was not commensurate with its demand. However, with the upsurge in biodiesel production, the glycerol availability increased tremendously because for every production of biodiesel, 10% by weight of it is glycerol (Quispe et al., 2013; Anitha et al., 2016). Available statistics shows that in the past years Europe and United States were the largest producers of glycerol with about 65% combined capacity but recent market research shows that the Asia-Pacific region has taken the lead to be the largest glycerol producer in the world with Malaysia and Indonesia been largest producer from palm oil (Christoph et al., 2012; Quispe et al., 2013).

With above scenario, the industrial accumulation of glycerol becomes a menace that needs to be addressed (Ahmad et al., 2021). Hence, researchers' attention have shifted to the conversion of glycerol to high value products or chemicals, otherwise known as repurposing (Karnjanakom et al., 2018). For a sustainable circular economy, it is important to consider repurposing glycerol obtained as a by-product of biodiesel production, to overcome the production cost and to also encourage biodiesel production in a wide range (Checa et al., 2020).

One of the promising means of repurposing glycerol into a valuable product is through transformation of glycerol via catalytic acetylation with acetic acid to yield mono-acetin (MA), diacetin (DA), and triacetin (TA) as illustrated in Figure 1.1 (Chong et al., 2020; Manríquez-Ramírez et al., 2020). These products of acetylation reaction have versatile applications in cosmetics, pharmaceuticals and fuel industry.

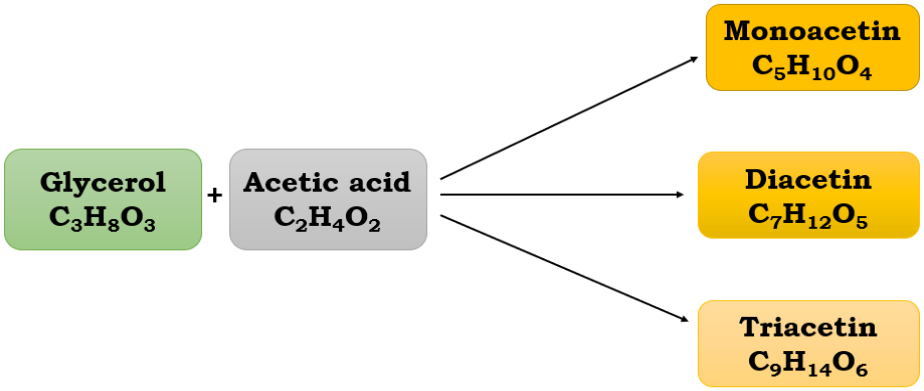

Figure 1.1: Illustration of glycerol conversion to monoacetin, diacetin, and triacetin

The need for a catalyst in a chemical reaction cannot be overemphasized, since it lowers the activation energy required to commence a reaction (Dhakshinamoorthy et al., 2021). This reason equips catalyst materials as a sustainable resource that can lower the temperature and time that will normally be taken to accomplish the desired reaction processes. Catalysts play a significant role in the chemical industry by promoting chemical reactions and also enhancing active selectivity of products and by-products, thereby minimizing reagent waste and undesired outcomes, eliminating or transforming pollutants into products that are considered non-toxic (Kumar et al., 2020a). Catalysts have gained much industrial prominence such that the global catalyst market was estimated to be worth $15 billion with about 95% of the industrial products produced via catalytic processes (O'Neill et al., 2015). Therefore, the deployment of appropriate catalyst (homogeneous or heterogeneous), in glycerol acetylation to yield the desired product(s) is necessary.

The application of homogeneous catalyst such as hydrochloric acid, hydrofluoric acid, sulphuric acid, or acidic ionic liquids in glycerol acetylation, though highly active, have since been limited due to some technical and environmental considerations, such as product purity, product separation, corrosion of reactor, non-recyclability, production of large amount of waste, adverse environmental impact, etc. Therefore, the task of developing efficient heterogeneous solid catalysts have become necessary to overcome the above disadvantages and provide better selectivity for the desired products. The use of so many heterogeneous acid catalysts such as heteropolyacids, amberlysts and zeolites are also characterized by certain defects such as thermal instability and poor regeneration ability hence the use of metal oxide catalysts in this study. The metal oxide catalysts are inexpensive and stable over a wide temperature range and easily regenerable (Sudarsanam et al., 2019). However, they have low surface areas and low pore size distribution hence the need to provide a good supporting material.

Among the catalyst supporting materials, carbon standout due to its unique physicochemical properties which include large surface area, tuneable pore structure,

chemical inertness, high electrical, thermal and mechanical stabilities (Goscianska and Malaika, 2020). Therefore, this study involves the derivation of carbon from a palm kernel shell (PKS) biomass and in turn use as a supporting material for a metal oxide (yttrium oxide) to serve as catalyst for glycerol acetylation. The application of PKS biomass as a component of a catalysts is an advantage towards its sustainable disposal route in the environment. In addition, its biodegradability and biocompatibility features offer wide arrays of opportunities for the development of functional and functional hybrid solids catalysts. The dawn of the new decade, encourages the scientific community towards studies that are focused on utilization of agro-wastes with a view to attaining efficiency and sustainability of resources. One of such utilization is biomass-derived catalyst or catalyst support for industrial applications.

1.2 Problem statement

Researchers have sought to continuously improve on the properties of heterogeneous catalysts that are suitable for an effective glycerol conversion into valuable products. The information on the use of metal oxide catalysts is still evolving and are therefore scanty but indicates their potentials in glycerol acetylation. However, they are limited by their low surface areas and low pore size distribution hence the need to provide them with adequate support materials. Therefore, this research was conceived in view of the earlier studies indicative of carbon as a good supporting material due to its excellent physicochemical properties, while yttrium oxide is thought of to be a good acid material. Since acetylation reaction strife under acidic catalyst with moderate surface area and adequate pore size distribution, a carbon support was generated from the abundant PKS and impregnated (functionalized) with yttrium oxide to provide the active sites, improve the surface area and pore size distribution of the resultant catalyst with a view to improving glycerol acetylation.

1.3 Significance of the study

The environment is becoming increasingly polluted in the 21st century. Both glycerol and palm kernel shells (PKS) are example of such major sources of pollution. Glycerol is a product of biodiesel which is increasingly being produced in Malaysia, while the palm kernel shells (PKS) constitute large scale biomass waste during palm oil production. They both constitute environmental nuisance due to large production and therefore, the need to repurpose them into value-added products for other uses. This can be achieved by utilizing the PKS as a carbon source while the glycerol can be converted to acetin, a very important and versatile industrial chemical. As such this research, is aim at converting waste to wealth thereby repurposing waste materials for value addition.

1.4 Research objectives

The objective of the study includes:

1. to generate carbon from palm kernel shell (PKS) and use it as a support material in the synthesis of carbon supported yttrium oxide catalyst.
2. to characterise the synthesized catalyst and test its activity in glycerol acetylation reaction.
3. to optimize the reaction conditions using response surface methodology.
4. to evaluate the reusability of the synthesized catalyst.

1.5 Scope of the study

In this study, carbon material was generated from palm kernel shell (PKS) using template carbonization method with the sodium silicate (Na_2SiO_3) as the templating agent. The generated carbon was used as support for the yttrium oxide (Y_2O_3) resulting in carbon supported yttrium oxide catalyst.

The synthesized catalysts and the precursor materials were characterized using Brunauer-Emmett-Teller (BET) technique, X-ray diffraction (XRD) technique, thermogravimetric analysis (TGA), temperature programme desorption of ammonia (TPD-NH_3), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX). The synthesized catalysts were evaluated in glycerol acetylation reaction with acetic acid. The glycerol conversion and the selectivity of the products were determined and used as the yardstick for the performance of the synthesized catalyst.

The reaction was optimized using the four-factor, two-level face-centred central composite design of the response surface methodology (RSM) with reaction temperature, reaction time, acetic acid mole ratio, and catalyst loading as the variables.
The reusability test of the catalyst was also studied by conducting acetylation reaction using the same catalyst in three reaction cycles. This also ascertained the catalyst degradation or otherwise of the catalyst.

1.6 Thesis outline

Chapter 1, dwells upon the research background, problem statement, the significance of the study, research objectives, and the scope of the research work.

Chapter 2, gives detail background about glycerol and biomass as a source of carbon material. It also delved into past and recent studies on the synthesis and application of various catalysts in glycerol acetylation and the influencing factors. It also discusses optimization principles and the types.

Chapter 3, deals with the materials and methods used for the research work. It also deals with the acetylation reaction and optimization methods used for the reaction as well as the various characterization methods used.

Chapter 4 is all about the results of the experiments and their discussions, while chapter 5, summarizes the study and provides future trends in the field of glycerol acetylation and other recommendations.

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LIST OF PUBLICATIONS

Nda-Umar, U. I., Ramli, I. B., Muhamad, E. N., Azri, N., **Amadi, U. F.,** & Taufiq-Yap, Y. H. (2020). Influence of Heterogeneous Catalysts and Reaction Parameters on the Acetylation of Glycerol to Acetin: A Review. *Applied Sciences*, *10*(20), 7155. MDPI AG. http://dx.doi.org/10.3390/app10207155

Amadi, U. F., Ramli, I. B., Taufiq-Yap, Y. H., Saimana, M. I., Usman Idris Nda-Umar, U. I. (2023). Glycerol acetylation over yttrium oxide (Y_2O_3) supported on palm kernel shell-derived carbon and parameters optimization studies using response surface methodology (RSM): *Arabian Journal of Chemistry*. 16(8), 104865. https://doi.org/10.1016/j.arabjc.2023.104865

List of Workshops/ Seminars/Conferences attended

1. The 12th International Fundamental Science Congress 2021 (iFSC2021 12th), 24th – 26th August 2021, Synthesis of Yttrium Oxide (Y_2O_3) Supported on Biomass-derived Carbon as a Catalyst for Glycerol Acetylation, Organised by Faculty of Science, Universiti Putra Malaysia. As Oral Presenter.

2. Information Literacy Programme : Online Data Base Training Session by Publisher, 11th April 2022, Organised by Sultan Abdul Samad Library, Universiti Putra Malaysia. As a Participant.

3. Theory and Data Analysis for Catalyst Characterization Workshop, 2nd February 2023, Organised by Dr. Mazni Binti Ismail (Deputy Dean of Postgraduate Research, faculty of Chemical and Process Engineering Technology. As a Participant.

4. Putra Plantation Forum Series-10, 9th December 2020, Biorefinary in the new Bioeconomy, Organised by Institute of Plantation Studies (IKP) Universiti Putra Malaysia. As a Participant.

5. Writing a Thesis to Prepare for Viva : Viva Imagination as a Tool for Thesis Preparation, 5th March 2021, Organised by Institute for Social Studies, Universiti Putra Malaysia. As a Participant.