UNIVERSITI PUTRA MALAYSIA

PREPARATION OF La_2O_3-CaO MIXED OXIDE CATALYSTS SUPPORTED ON NANOSTRUCTURED ACTIVATED CARBON FOR BIODIESEL PRODUCTION FROM WASTE COOKING OIL

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October 2016

Chairman : Professor Taufiq Yap Yun Hin, FASc, PhD
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Advanced carbon nanorod promoted binary La_2O_3-CaO system with improved physical properties, tailored surface morphology and chemistry were developed in vacuum-impregnating methods. The nanostructured catalyst (CaO–La_2O_3/AC nanocatalyst) was prepared to convert waste cooking oil with high FFA into biodiesel via one step esterification-transesterification reaction. The novel catalyst was characterized by XRF, FTIR, HRTEM, FESEM, XRD, TGA, BET, TPD-CO_2 and TPD-NH_3. The high catalytic activity of the nanocatalyst was mainly depended on the high acid and basic density of active sites that contributed from the synergic effect between mesoporous carbon and binary metallic system, which allowed more occurrence of simultaneous esterification-transesterification process of high FFA waste oil without additional pretreatment step. Results showed a maximum triglyceride conversion of 98.6 ± 0.5% with acid value 0.4 ± 0.5 mg KOH/g of triglyceride conversion under optimal condition at 3% of catalyst, methanol: oil ratio of 16:1, 100 ^{o}C within 4 h of reaction. Furthermore, bi-metallic catalysts with stable carbon nanorod support were capable to maintained high reusability with high FAME yield (> 98%) with low acid value (<0.5 mg KOH/g) for 5 cycles. Several physicochemical properties of WCO-based biodiesel produced were tested and in good agreement to ASTM D6751 standards.

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

PENYEDIAAN PEMANGKIN OKSIDA CAMPURAN La_2O_3-CaO YANG DISOKONG ATAS KARBON YANG DIAKTIFKAN DAN BERNANOSTRUKTUR UNTUK PENGELUARAN BIODIESEL DARIPADA SISA MINYAK MASAK

Oleh

ABDULKAREEM GHASSAN ABDULKREEM

Oktober 2016

Pengerusi : Profesor Taufiq Yap Yun Hin, FASc, PhD
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Sistem binari CaO-La_2O_3 yang digalakkan oleh nanorod karbon yang maju, dengan ciri-ciri fizikal yang lebih baik and morfologi permukaan dan kimia yang disesuaikan, telah dibangunkan dengan menggunakan kaedah resapan vakum. Pemangkin bernanostruktur (CaO–La_2O_3/AC pemangkin nano) disediakan untuk menukar sisa minyak masak yang tinggi FFA kepada biodiesel melalui tindakbalas selangkah pengesteran-transesterifikasi. Pemangkin baru tersebut dicirikan oleh XRF,FTIR,HRTEM, FESEM, XRD, TGA, BET, TPD-CO_2 dan TPD-NH_3. Aktiviti bermangkin tinggi pemangkin nano tersebut banyak bergantung kepada keasidan dan kepadatan asas yang tinggi pada tapak aktif yang menyumbang daripada kesan bersinergi antara karbon berliang meso dan sistem logam binari, yang membolehkan lebih banyak berlakunya proses pengesteran-transesterifikasi serentak terhadap sisa minyak dengan FFA tinggi tanpa tambahan langkah rawatan awal. Keputusan menunjukkan maksimum sebanyak 98.6 ±0.5% dengan nilai asid 0.4 ±0.5 mg KOH/g dalam penukaran trigliserida di bawah keadaan optimum pada 3% daripada pemangkin, dengan nisbah metanol:minyak sebanyak 16:1, pada suhu 100 ºC dalam masa tindakbalas 4 jam. Tambahan pula, pemangkin bi-logam dengan sokongan nanorod karbon yang stabil mampu mengekalkan kebolehan gunapakai tinggi dengan alah FAME yang tinggi (> 98%) dengan nilai asid yang rendah (<0.5 mg KOH/g) untuk 5 kitaran. Beberapa ciri-ciri fizikokimia biodiesel berasaskan WCO yang diuji adalah bertepatan juga dengan piawai ASTM D6751.

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I certify that a Thesis Examination Committee has met on 5 October 2016 to conduct the final examination of Abdulkareem Ghassan Abdulkareem on his thesis entitled "Preparation of La_2O_3-CaO Mixed Oxide Catalysts Supported on Nanostructured Activated Carbon for Biodiesel Production from Waste Cooking Oil" 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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LIST OF ABBREVIATIONS AND SYMBOLS

ASTM	American society for testing and materials
AC	Activated carbon
WCO	Waste cooking oil
EN	European Standard
ATZ	5-AMINOTETRAZOLE-HYDRATE
ATB	6-methoxy-α-methyl-2-naphthaleneacetic acid, 4-(aminothioxomethyl)phenyl ester
BET	Brunauer-Emmett-Teller
EDX	Energy-dispersive X-ray spectroscope
FAME	Fatty acid methyl ester
FFA	Free fatty acid
FT-IR	Fourier transform infrared
GC	Gas chromatograph
GC-MS	Gas chromatograph mass spectrometry
HRTEM	High-resolution TEM
TPD- NH_3	Ammonia-temperature programmed desorption
TPD-CO_2	Carbon dioxide- temperature programmed desorption
FESEM	Field emission scanning electron microscope.
TGA	Thermogravimetric analysis
XRD	X-ray power diffraction

CHAPTER 1

INTRODUCTION

1.1 Catalyst

The word 'catalysis' derives from the Greek word meaning 'to dissolve'. A catalyst is typically defined as a substance which enhances the rate at which a chemical reaction proceeds by reducing the activation energy required (Figure 1). It is able to act in very small quantities as an initiator for the transformation and is not used up itself in the process (Ertl *et al*., 2008). Ertal *et al*. (2008) also refer to Laidler (1986) Ostawld's (1895), the alternative definition of a catalyst is a substance which is able to enhance a chemical reaction without affecting its equilibrium.

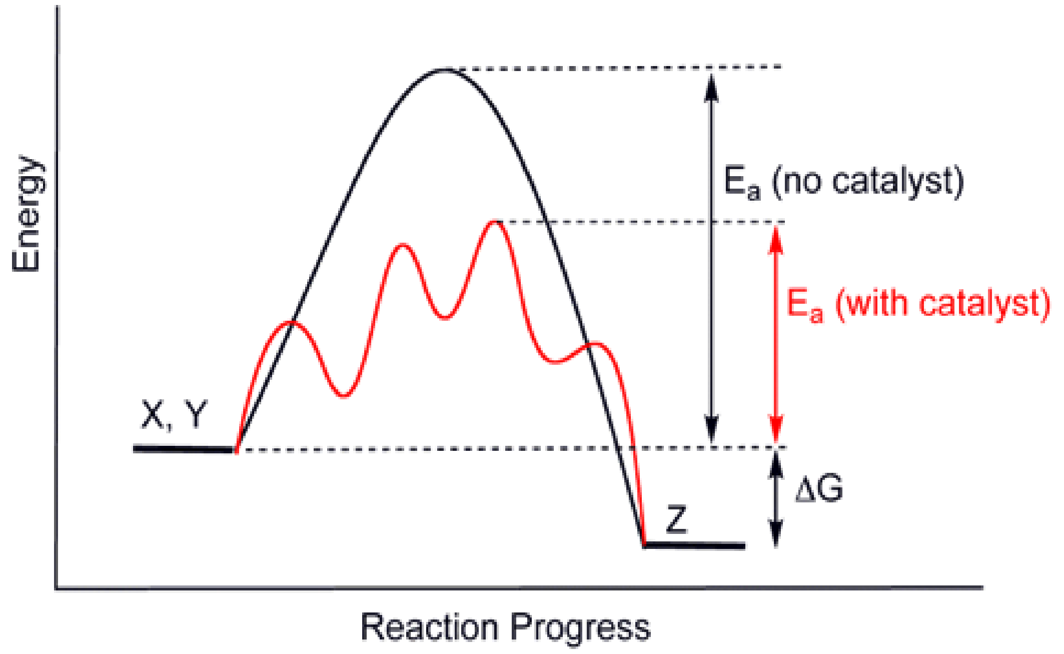

Figure 1 : Illustration of the effect of a catalyst in reducing the activation energy required for the exothermic reaction X + Y —> Z (Robertson, 1970).

Since catalysis represents a means for facilitating chemical reactions with lower energy requirements, it can therefore be viewed as a process with environmental as well as commercial benefits. In addition, it also offers two significant technical advantages: selectivity and thermal stability which again contribute to improved outcome for environmental or 'green' considerations.

Roebuck in 1746, whilst producing sulphuric acid was the first to use a catalyst — a development which the chemical industry quickly picked up with wide application.

Initially, only pure elements were utilised a catalysts, however, from the start of the twentieth century, compound catalysts were developed and are widely deployed today (Leach, 1983). Examples of major industrial applications utilising the process of catalysis include those for the production of ammonia and methyl alcohol. Both these processes involve state change of reactants facilitated by solid catalysis. According to Robertson (Robertson, 1970), around 85–90 % of industrial chemical processes involve some form of catalysis, with the final products valued at around $900bn worldwide.

1.2 Catalysts for Energy

Increasing population growth, economic growth and energy demand has led to concern regarding limited energy resources, particularly with regard to the overconsumption of non-sustainable fossil fuel energy sources which is now known to be responsible for global warming and climate change. This situation represents major environmental and economic challenges facing the world today and in the future. Therefore, renewable forms of energy have been proposed as a way forward, and catalysis represents renewable processes that can reduce energy requirements for chemical reactions. Furthermore, new technology is starting to examine the possibility of utilising catalysts for the production of greener and clean energy, which by its application able to reduce greenhouse gas emissions.

1.3 Background

Since the mid-nineteenth century, fossil fuel has been the most significant and easily available energy source in the world. Today around 90% of all vehicles are powered by fossil fuel. It is well known that petroleum products provide major source materials for the chemical industry producing plastics, pharmaceuticals, pesticides, fertilizers and solvent (Simanzhenkov *et al.*, 2003; Speight *et al.*, 2001). However, the non-renewable nature of the fossil fuel source materials has become a growing cause for concern from the latter half of the twentieth century onwards. Demand for sustainable, renewable energy sources has grown as people have sought to decrease their dependency on fossil fuels and their associated uncertainties of security of supply and price variability. Polluting gaseous emissions from burning fossil fuels are another factor driving the search for a clean energy supply (Mizsey *et al.*, 2010). Biofuel and biomass technologies have the potential to provide significant sources of energy in the next century.

Biofuel, is an alternative cleaner burning fuel that successfully been tried and used by majority of the nation to replace the use of non-sustainable fossil energy. Biofuel can be referred as liquid or gaseous fuels use in transportation sector which predominantly synthesized from biomass. Therefore, application of biofuel have been proved to be meeting wide range of social and environmental sustainable criteria including limit on deforestations, competition with food production, adverse impact on biodiversity, soil erosion and nutrient leaching. Biofuels are derived from renewable sources and include biodiesel, green diesel, bioethanol, biogas, biomethanol, synthetic biofuel and biohydrogen(Justine *et al.*, 2009). Currently, bioethanol and biodiesel account for around 90% of the biofuel market.

1.4 Biodiesel

Biodiesel is considered a viable alternative to petroleum–derived fuel. Chemically, biodiesel consists of a methyl ester with long chain fatty acids (FAME) derived from transesterification or esterification reaction of lipid feedstocks such as vegetable oils or animal fats(Narayan & Madras, 2016) and (Monirul *et al.*, 2015). Biodiesels are renewable, biodegradable, less toxic, have an environmentally friendly emission profile, a higher combustion efficiency, higher certain number, higher flash point and better lubrication than petroleum derived fuel (Alsultan *et al.*, 2016; Can *et al.*, 2016). Moreover, biodiesel and diesel share similar physicochemical properties (Table 1), so biodiesel can be used on its own or mixed with diesel in conventional compression ignition engines with some engine modifications.

Table 1 : Comparison of biodiesel and diesel according to the American Standard for Testing and Materials (ASTM)

Property of the fuel	Biodiesel	Diesel
Standard method	ASTM D6751	ASTM D975
Fuel composition	FAME(C_{12}-C_{22})	Hydrocarbon(C_{10}-C_{21})
Density(g/cm^3)	0.878	0.848
Pour point (^{o}C)	-15 to 16	-30 to -15
Cloud point(^{o}C)	-3 to 12	-15 to 5
Flash point(^{o}C)	100-170	60-80
Cetane number	48-60	40-55
Water (vol %)	0.05	0.05
Carbon (wt. %)	77	87
Hydrogen (wt. %)	12	13
Oxygen (wt. %)	11	0
Sulphur (wt. %)	0.05	0.05

Biodiesel is considered the best candidate for petroleum derived fuel substitute in diesel engine (compression-ignition engines) due well known advantages, including following (Zhong *et al.,* 2016). Biodiesel has good combustion properties due to its high oxygen content (11%). The Lifecycle analysis indicates that biodiesel has 78% lower net carbon dioxide emissions than conventional diesel and has a lower smoke emission factor as a consequence of minimal free soot.in addition, Biodiesel is less damaging to the environment than conventional diesel since it is biodegradable, sustainable, renewable, non-toxic and has lower sulphur content and the claimed to reduce incidence of dangerous disease by 90%. Biodiesel also offers the potential of various socioeconomic advantages such as employment, investment in rural areas and reduced negative consequences of global warming. Biodiesel has a higher cetane value than conventional diesel. The production of biodiesel is easier to produce than conventional diesel. And Biodiesel is a better lubricant than conventional diesel and therefore a better promoter of engine efficiency. Biodiesel has a higher flashpoint than conventional diesel (Table 1.1) which is a safety feature, reducing likelihood of explosive combustion. Biodiesel offers the additional environmental benefit with the potential to recycle food oils, removing them from the waste stream. Biodiesel (B20) in low blends can be used directly (without modification) in engines, although some engine modification may be required to use higher ratios of biodiesel.

1.5 Problem Statement

Typical feedstock used for biodiesel production mainly derived from edible oil that available abundantly around the world. These source materials have led to food vs fuel concerns as conflicts between the needs for biofuel and human food have been reported. The competition between food and fuel economics toward the same oil might bring global imbalance to the food supply and demand on the market. In addition, utilization of edible oil also surely will lead to higher production cost which unattractive for industrial scale Thus, in order to commercially viable alternative to petroleum derived fuel industry, the use of lower-cost and non-edible oil such as waste cooking oil (WCO) is taken into consideration in biodiesel production.

Nowadays, there are a lot of heterogeneous solid catalysts that have been introduced for biodiesel applications due to easy purification step and high reusability as compared to homogeneous catalysts. Different type of solid catalysts was proposed, such as base (alkaline-based and alkaline-earth-based metal oxide) and acid (functionalization of silica materials (MCM-41 and SBA-15) with organo-sulfonic acids) catalysts. Typically, transesterification catalyzed by solid base catalysts show high reactivity under the mild reacting condition as compared to solid acid catalysts. However, the WCO is an acid oil with large amounts of free fatty acids (FFAs), which the active site of base catalyst for transesterification reaction was normally inhibited by the fatty acid via saponification. Ideally, acid catalysis is a potential candidate for simultaneous esterification of the FFAs and transesterification of the triglycerides to achieve one-pot preparation of FAMEs from WCO. Therefore, heterogeneous solid acid catalyst provides an environmentally benign and cost effective process for production of biodiesel from low quality acid oil. However, challenge on developing an efficient solid acid catalyst for one-step esterification-transesterification was still on-going.

In the biodiesel production, the most important to improve the process efficiency is the catalyst. However, heterogeneous catalyst was used instead of homogeneous catalyst because of the separation and corrosion problems. On the other hand, the catalytic activity and the stability of the heterogeneous catalyst are the key factors in synthesizing a novel catalyst. To overcome this matter, the carbon-based solid acid catalysts were introduced in this research, which had proved to have high catalytic activity and good stability.

1.6 Objectives

The objectives of this study are:

1. To synthesis and characterization nanosize solid acid-base catalysts (La_2O_3-CaO/AC) to facilitate the manufacture of biodiesel from waste cooking oil.
2. To determine the catalytic activity and influential factors in esterification-transesterification process of synthesized acid-base catalysts.
3. To study the reusability and catalyst stability in the esterification-transesterification process.

1.7 Scope of Study

Advanced carbon nanorod promoted binary La_2O_3-CaO system with improved physical properties, tailored surface morphology and chemistry were developed in vacuum-impregnating methods. This nanosize mixed metal oxides supported activated carbon derived walnut shell (*juglans sp*.) are believed to consist of superior physicochemical properties than activated carbon support and enhance the one-step esterification-transesterification process of waste cooking oil. The mixed metal oxides supported on AC with in cooperation of La metal content from 5 to 30% toward one-step esterification-transesterification activity were further investigated. The highest catalytic activity from mixed metal oxides supported AC with optimum ration of Ca metal was selected and further optimize in one-step esterification-transesterification process. Besides, the reusability and catalyst stability also were studied.

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