SYNTHESIS AND CHARACTERIZATION OF MOLYBDENUM CARBIDE FROM OIL PALM FROND BASED ACTIVATED CARBON FOR CARBON DIOXIDE REDUCTION

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For my beloved family & friends………

To my beloved family that never stopped supporting, guiding and believe in countless ways, both direct and indirect. I was going to start listing them all, but realized they are just too many to do that justice - so please accept the fact that you are all mentioned in my daily prayer of thanks to a loving ALLAH s.w.t. Who will convey that thanks in His own way back to you.

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ABSTRACT

Energy-efficient technique is one of the vital keys to sustainable development in manufacturing processes. Microwave technique is an attractive approach in manufacturing as this technique offers several advantages such as rapid process and low energy requirement. In this study, microwave induced technique was applied to the preparation of activated carbons and molybdenum carbides. The activated carbons were prepared using oil palm fronds and activated using potassium hydroxide and phosphoric acid solutions at different concentrations. The products were then characterized using Fourier transform infrared spectroscopy (FTIR), nitrogen gas adsorption analysis and scanning electron microscopy (SEM). The results indicated that the highest surface area obtained by activation using phosphoric acid was 638.14 m^2/g. However, the activated carbons prepared using a potassium hydroxide showed low surface areas for all samples. In the production of molybdenum carbide and potassium doped molybdenum carbide via microwave induced alloying, the prepared activated carbons were mixed with a 1 M molybdenum solution followed by the microwave-induced technique. All the prepared samples were chemically characterized using X-ray diffraction analysis (XRD), nitrogen gas adsorption analysis, SEM, energy dispersive X-ray (EDX), ammonia-temperature-programmed desorption analysis (TPD-NH_3), acidic titration method and electron spin resonance (ESR) spectroscopy. XRD analysis indicated that the perfect ratio for the preparation of molybdenum carbide is 2 Mo: 7 C. Nitrogen gas analysis showed that molybdenum carbide and potassium doped molybdenum carbide had surface areas of 95.0537 and 61.9302 m^2/g, respectively. The ESR spectroscopy indicated the presence of a singlet peak at g-value of 1.9723 in the ESR spectra of potassium doped molybdenum carbide; this suggested that an electron was donated from potassium to the surface of molybdenum carbide. TPD-NH_3 indicated the presence of weak, moderate and strong acid sites for the molybdenum carbide and potassium doped molybdenum carbide. However, the addition of potassium decreased the amount of acid sites for the molybdenum carbide. The prepared carbides were evaluated as catalysts in the reduction reactions of carbon dioxide that were conducted using in-house modified reactor. The product was then characterized using FTIR. In the catalytic application, the molybdenum carbide showed a higher activity compared to the potassium doped molybdenum carbide. This was probably caused by the potassium moiety which poisoned the reaction while blocking the active sites. To better understand the reaction, kinetic analysis was carried out for the hydrogenation of carbon dioxide via molybdenum carbide. Results suggested an optimum temperature of 450°C, second order reaction, and the activation energy of 34.27 kJ/mol. In addition, thermodynamic parameters were also calculated using the Eyring equation and it was revealed that the values of $\Delta H^{\ddagger}$, $\Delta S^{\ddagger}$ and $\Delta G^{\ddagger}$ were +40.04 kJ/mol, -1478.4 J/mol K and +1108.7 kJ/mol, respectively. These parameters proved that this reaction is not spontaneous and it is endothermic in nature with associative reaction mechanism.

ABSTRAK

Teknik cekap tenaga adalah salah satu kunci penting untuk pembangunan lestari dalam proses pembuatan. Teknik gelombang mikro merupakan pendekatan yang menarik kerana teknik ini menawarkan beberapa kelebihan seperti proses yang pesat dan keperluan tenaga rendah. Dalam kajian ini, teknik gelombang mikro teraruh telah digunakan untuk menyediakan karbon diaktifkan dan molibdenum karbida. Karbon-karbon teraktif telah disediakan dengan menggunakan pelepah kelapa sawit dan diaktifkan dengan menggunakan larutan kalium hidroksida dan asid fosforik pada kepekatan berlainan. Produk-produk kemudiannya dicirikan menggunakan spektroskopi inframerah transformasi Fourier (FTIR), analisis penjerapan gas nitrogen dan mikroskopi imbasan elektron (SEM). Keputusan menunjukkan bahawa luas permukaan yang paling tinggi yang diperolehi dengan pengaktifan menggunakan asid fosforik ialah 638.14 m^2/g. Walau bagaimanapun, karbon diaktifkan yang disediakan menggunakan kalium hidroksida menunjukkan luas permukaan yang rendah untuk semua sampel. Dalam penyediaan molibdenum karbida dan molibdenum karbida terdopkan kalium melalui pengaloian gelombang mikro teraruh, karbon diaktifkan yang telah disediakan dicampur dengan larutan molibdenum 1 M diikuti oleh teknik gelombang mikro teraruh. Semua sampel yang telah disediakan dicirikan secara kimia menggunakan analisis pembelauan sinar-X (XRD), analisis penjerapan gas nitrogen, SEM, serakan tenaga sinar-X (EDX), analisis suhu yang diprogram penjerapan gas ammonia (TPD-NH_3), kaedah titratan berasid dan spektroskopi resonans spin elektron (ESR). Analisis XRD menunjukkan bahawa nisbah yang sempurna untuk menyediakan molibdenum karbida ialah 2 Mo: 7 C. Analisis gas nitrogen menunjukkan karbida molibdenum dan molibdenum karbida terdopkan kalium mempunyai luas permukaan masing-masing 95.0537 dan 61.9302 m^2/g. Spektroskopi ESR menunjukkan kehadiran puncak tunggal di nilai-g 1.9723 bagi molibdenum karbida terdopkan kalium; ini mencadangkan elektron disumbangkan daripada kalium ke permukaan molibdenum karbida . Analisa TPD-NH_3 menunjukkan kehadiran tapak asid yang lemah, sederhana dan kuat bagi molibdenum karbida dan kalium terdopkan molibdenum karbida. Walau bagaimanapun, penambahan kalium telah mengurangkan jumlah tapak asid untuk molibdenum karbida itu. Karbida-karbida yang telah disediakan dinilai sebagai pemangkin di dalam tindak balas penurunan karbon dioksida yang telah dijalankan menggunakan reaktor diubahsuai di dalam makmal. Produk yang terhasil kemudiannya dicirikan menggunakan FTIR. Dalam aplikasi bermangkin, karbida molibdenum menunjukkan aktiviti yang lebih tinggi berbanding dengan molibdenum karbida terdopkan kalium. Ini mungkin disebabkan oleh moieti kalium telah meracuni tindak balas, pada masa yang sama menghalang tapak aktif. Untuk lebih memahami tindak balas, analisis kinetik telah dijalankan untuk penghidrogenan karbon dioksida melalui molibdenum karbida. Keputusan mencadangkan suhu optimum adalah 450°C, tindak balas peringkat kedua, dan tenaga pengaktifan kira-kira 34.27 kJ/mol. Tambah lagi, parameter termodinamik juga telah dikira dengan menggunakan persamaan Eyring dan didapati bahawa nilai $\Delta H^‡$, $\Delta S^‡$ dan $\Delta G^‡$ adalah masing-masing +40.04 kJ/mol, -1478.4 J/mol K dan +1108.7 kJ/mol. Parameter-parameter ini membuktikan bahawa tindak balas ini tidak spontan dan endotermik secara semula jadi dengan mekanisma tindak balas yang bersekutu.

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LIST OF SYMBOLS

°C	-	degree Celsius
μm	-	micrometer
nm	-	nanometer
cm	-	centimetre
g	-	gram
ID	-	internal diameter
K	-	Kelvin
mL	-	milliliter
θ	-	Half angle of diffraction beam
λ	-	Wavelength

LIST OF ABBREVIATIONS

BET	-	Brunauer-Emmett-Teller
DTA	-	Differential thermal analysis
TGA	-	Thermogravimetry analysis
EDX	-	Energy Dispersive X-ray analysis
DFT	-	Density Functional Theory
FTIR	-	Fourier Transformation Infrared
ESR	-	Electron Spin Resonance
TPD	-	Temperature Programmed Desorption
SEM	-	Scanning Electron Microscope
NA	-	nitrogen adsorption
XRD	-	X-ray Diffraction
FESEM	-	Field Emission Scanning Electron Microscope
XRD	-	X-Ray Diffraction
KBr	-	Potassium Bromide
TMC		Transition Metal Carbide
MIA		Microwave Induced Alloying
AHM		Ammonium Hepta Molybdate

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CHAPTER I

INTRODUCTION

1.1 Background of Research

The future of the materials, manufacturing technology depends on three major ideas; energy efficiency, sustainability and economic viability. Among of these ideas, energy efficiency is the top priority for the growth of this industry as researches spend a lot of time and energy to find and introduce any new energy efficient manufacturing technology as a conventional heating method waste a huge amount of energy, therefore affected the environment. Introduction of microwave technique nowadays seems like a promising solution as this technology offers several advantages such as rapid processing, energy efficient process and reduced cost of operation. In addition, the microwave method also opens new edge of knowledge, especially on the interaction of this energy to the atomic structure of the starting material which could be a reason for the rising interest. Since the first report of this technology in 1971, significant and positive rising numbers of publications were shown for the past few decades. Thus, in this research, application of microwave heating was fully utilized, especially in the preparation of activated carbon and molybdenum carbide.

Recently, microwave induced activation or microwave heating has been introduced in the preparation of activated carbon. This new technology is applied as the carbonaceous material known as a microwave absorbent. Beside faster time of preparation and energy efficient method, another reason could be the intention of selecting this kind of heating method is the prepared activated carbon has a higher surface area and pore volume (Tan *et al.*, 2008). These two properties are highly required for the best activated carbon, therefore, microwave energy is proven to achieve this result. Thus, in this study, the activated carbon was prepared via Microwave Induced Activation technique using waste oil palm frond as a carbon source and two activation agents, which are potassium hydroxide and phosphoric acid respectively.

A number of methods were proposed for molybdenum carbide synthesis and each process has its own characteristics of powder produced; Combustion Synthesis (CS), Self-propagating High Temperature Synthesis (SHS), Direct Carburization, Field Activated combustion method. However, the major drawback of these methods is it requires long time of combustion and high energy preparation. Thus, many researchers suggested the easier, faster and energy saving ideas for transition metal carbides synthesis and microwave looks promising. (Lu *et al.*, (2012) had successfully prepared tungsten carbide/ carbon composite by the Microwave Heating (MH) technique. This method offers numerous advantages compared to other access method, for instance, it can be rapidly synthesized in one-step process, and thus it is economical and can be synthesized at lower temperature. In addition, Kitchen *et al.*, (2014) underlined that strong coupling of carbon with microwave is the key of success of this application. Thus, in this research, the molybdenum carbides were prepared via Microwave Induced Alloying.

TMC especially Mo_2C were studied for their reactivity on oxidation, hydrogen transfer reaction such as hydrogenation, isomerization, desulfurization and methanation. In this case, many researches proved that the catalytic behavior of TMCs matched and some time, exceeding the best-known precious noble metal in hydrogen transfer reaction. Compared to metal sulfides, metal carbide possesses superior hydrogen adsorption, activation and transfer capabilities, and then it has prospective application in CO hydrogenation reactions (Fang *et al.*, 2009). Besides that, molybdenum carbide is also active in various reactions, such as decomposition of formic acid (Flaherty *et al.*, 2010), water gas shift (WGS) react to produce hydrogen (Moon, 2009) and hydrogenation reaction (Liu *et al.*, 2014, Xu *et al.*, 2014, Pang *et al.*, 2012 and Aoki *et al.*, 2013).

Carbon dioxide, known as greenhouse gas, mainly generated from the combustion reaction of organic compound. Previously, this gas always considers as waste product as well as contributing to global warming, however, this image has turned drastically as great resources, plus business opportunity since researcher found that recycling of CO_2 via catalytic conversion reaction is the promising method to reduce total contents of CO_2 in the atmosphere and generate valuable product such as methane and methanol (Centi & Perathoner 2004, De Falco *et al.*, 2013, Wang & Gong 2011 and Kaiser *et al.,* 2013). On the other hand, the recycling idea opens new dimensions of research and technology regarding catalyst to handle this reaction perfectly.

In this study, Microwave Induced Alloying (MIA) is introduced using simple modified household microwave oven to prepare the activated carbon from the waste oil palm frond and also, molybdenum carbide and potassium doped molybdenum carbide. In addition, this study also explores the effect of potassium as a dopant to the properties of the molybdenum carbide as well as for the reaction. Several literature papers suggested the potassium as a dopant initiated significant influence to the surface and catalytic properties of the molybdenum carbide (Chiang *et al.,* 2012, Kotarba *et al.,* 2004 and Pistonesi *et al.,* 2012). Therefore, for the preparation of potassium doped molybdenum carbide, the unwashed activated carbon prepared via potassium hydroxide were used as this preparation tends to manipulate the potassium residues which always remained as impurities in the preparation of activated carbon.

Consequently, the best prepared molybdenum carbide and potassium doped molybdenum carbide were applied as a catalyst for the hydrogenation of CO_2. Catalyst performance in terms of capability and reactivity to convert CO_2 become the Key Performances Index (KPI) to evaluate the quality of this catalyst, thus hopefully contribute to knowledge's world.

1.2 Research Problems

This research is purposely to introduce another edge of knowledge for the preparation of the molybdenum carbide. To date, the widespread method to prepare the molybdenum carbide is the temperature-programmed reduction using hydrocarbon as a carbon source. However, Liang *et al.*, (2002) explained that this technique has several drawbacks such as high temperature of preparation, the carburization processes must be carefully controlled, prepared carbide surface is contaminated by polymeric carbon and furthermore blocks and covers the active site. Thus, to elucidate this problem, the application of the activated carbon has been approached as a carbon source for this research. Activated carbon generally has a high surface area and well-developed pores might help in term of enhancing the surface area of the product as well as absorption capability. These two characters are obliging for this catalyst, especially this catalyst was tested to the gaseous reaction.

Secondly, there is a limited number of the published paper regarding the kinetic and thermodynamic for the carbon dioxide hydrogenation via molybdenum carbide as a catalyst. Thus, this fact opens the opportunity for this study to introduce and suggest new ideas to understand and explore this area. The majority of the published literature discussed more specific to the activity of the catalyst and proposing a kinetic model to represent their work. However, no specific links to incorporate the kinetic and thermodynamic of the reaction as these two subjects are supposed to be a connected. As the kinetic purposely to study the effectiveness of the catalyst, the thermodynamic approach is compulsory to prove this reaction is workable and conducted by the catalyst.

1.3 Research Objectives

The objectives of this research are listed below:

1. To prepare and characterize the activated carbon via Microwave Induced Alloying technique.
2. To prepare and characterize molybdenum carbide and potassium doped molybdenum carbide via Microwave Induced Alloying technique.
3. To determine the activity of nano molybdenum carbide and potassium doped molybdenum carbide in the CO_2 hydrogenation reaction.
4. To study the mechanism of CO_2 hydrogenation reaction catalyzed by prepared nano molybdenum carbide and potassium doped molybdenum carbide.
5. To analyze the kinetic and thermodynamic parameters of carbon dioxide hydrogenation via the prepared molybdenum carbide.

1.4 Research Scope

The scope of the study is divided into 4 major stages. The first stage is the preparation of activated carbon using waste oil palm frond. This stage introduced Microwave Induced Activation technique to prepare the activated carbon. At this stage, two different sources of activating agents were used, which were potassium hydroxide and phosphoric acid respectively. The prepared activated carbons were characterized using Fourier transform infrared, nitrogen gas adsorption analysis and field emission scanning electron microscope –energy dispersive X-ray.

The second stage of this research is the preparation of molybdenum carbide and potassium doped molybdenum carbide. For this stage, ammonium hepta molybdate was used as a metal precursor while the prepared activated carbons plays role as carbon sources. Selection of the activated carbon was based on highest surface area obtained by nitrogen gas adsorption analysis. Microwave induced alloying technique was introduced to substitute the conventional carburization process of preparation molybdenum carbide. For the potassium doped molybdenum carbide, the prepared potassium doped activated carbon was applied as a carbon source, which was previously activated using the known amount of the potassium hydroxide. The prepared carbon with the highest amount of potassium was applied as a carbon precursor for the preparation of potassium doped molybdenum carbide. The physicochemical properties of the prepared molybdenum carbide and potassium doped molybdenum carbide was characterized via X-ray diffraction analysis, scanning electron microscope (SEM), temperature programmed desorption, acidic-titration method, electron spin resonance and nitrogen gas adsorption Analysis.

The third stage of this research is the prepared molybdenum carbide and potassium doped molybdenum carbide were applied as a catalyst for CO_2 hydrogenation. The main objectives are to find out the activity, selectivity and stabilities of the prepared molybdenum carbide and potassium doped molybdenum carbide as a catalyst for this reaction. Furthermore, the mechanism of this reaction was proposed at the end of this project.

Lastly, the kinetic and thermodynamic parameters for hydrogenation of carbon dioxide were analyzed. The analysis of kinetic employed basic graphical method to identify the reaction order and rate. In addition, the activation energy was also calculated using the Arrhenius Law. Then, for the thermodynamic analysis, the acknowledged equations, Eyring equation and van't Hoff equation were used to calculate the thermodynamic parameters which are entropy, enthalpy and the Gibbs free energy of activation.

1.5 Significance of Research

This study expectantly can contribute to the knowledge's world by applying the Green Chemistry principle as a main inspiration to conduct this research. To date, in the preparation of materials, researchers nowadays always find the right technique to prepare their material at the same time, try to minimize the energy required. Generally, the main problem of the heat source from the conventional oven is the heat loss to the environment could make the reaction requires more time for the heat transfers. Thus, in this research, the application of the microwave energy purposely to counter this problem as the microwave energy offers a better energy penetration and minimizes the loss of energy to the environment.

Carbon dioxide is one of the greenhouse gases which is produced from complete combustion of fuel normally comes from engines in transportations and generator. This gas is may pollute the environment by creating a heat-trapping layer at the atmosphere and cause the global warming. Furthermore, Malaysia was declared at Copenhagen Climate Change Summit, 2008 to reduce up to 40% of total carbon dioxide emissions. In order to reduce the content of this gas in the environment, it has to be converted to form methane using molybdenum carbide as a catalyst prepared from the most abundant solid waste generated from palm oil processing mills via Sabatier synthesis. Application of molybdenum carbide (Mo_2C) as catalyst in the hydrogenation reaction is a good substitute for the more expensive platinum of palladium noble metals.

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