DEVELOPMENT OF HIGHLY EFFICIENT NICKEL-DOPED ZEOLITIC IMIDAZOLE FRAMEWORK (ZIF-67) BASED CATALYST FOR METHANATION REACTION

By

ALDOGHACHI ALI FARIS ABDULRIDHA

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

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

DEVELOPMENT OF HIGHLY EFFICIENT NICKEL-DOPED ZEOLITIC IMIDAZOLE FRAMEWORK (ZIF-67) BASED CATALYST FOR METHANATION REACTION

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ALDOGHACHI ALI FARIS ABDULRIDHA

May 2023

Chairman : Sivasangar a/l Seenivasagam, PhD
Faculty : Science

The conversion of CO_2 to synthetic natural gas via methanation reaction is gaining popularity due to its potential to solve long-term energy storage challenges and reduce CO_2 emissions. Developing a highly efficient catalyst that can actively function under low reaction temperatures is crucial for the methanation reaction's sustainability and for making CO_2 capture more affordable. Utilizing highly porous metal-organic framework (MOF) materials such as ZIF-67 as catalyst support with added Ni would be an interesting endeavor to achieve good dispersion and resist agglomeration of dopants during methanation reaction. Therefore, ZIF-67 was prepared using 2-methylimidazole and cobalt nitrates, and the obtained ZIF-67 solid was activated under a vacuum oven overnight at 80 °C. Nickel dopant was incorporated onto ZIF-67 in the range of 1-12 wt% via wet-impregnation method, and the catalyst precursor was calcined at 350 °C for 4h under N_2 flow. The prepared catalysts were characterized using Brunauer-Emmett-Teller (BET), X-ray diffraction (XRD), Thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), Temperature-programmed desorption of carbon dioxide (TPD-CO_2), Temperature-programmed reduction (TPR), and High-resolution transmission electron microscopy (HRTEM). Prior to the methanation reaction, 0.05g of catalyst was loaded into a fixed tubular reactor and reduced in situ under (5% H_2/balanced Ar) flow at 350 °C for 3h. Subsequently, the flow is switched to reactant gas for catalytic testing at a predetermined reaction temperature. Preliminary catalytic methanation reaction was carried out at a temperature of 400°C, with a flow rate of 108,000 $mL.g^{-1}.h^{-1}$ and feedstock gas (H_2/CO_2) ratio of 4:1. The physicochemical characterization of the prepared catalysts exhibit very high surface area and porosity along with evenly distributed Ni dopants over ZIF-67 support. The catalysts were found to be active in CO_2 conversion, whereby the presence of Ni significantly influences the product selectivity of the reaction. Based on the catalytic screening results, 8wt% Ni/ZIF-67 catalyst was deemed the most active among the other prepared catalysts in the methanation reaction, with a moderate CO_2 conversion of 56% followed by selectivity of 96% at a reaction temperature of 300°C.

Furthermore, the catalytic activity appeared to be highly stable, with a conversion of 53% and product selectivity of 94% for 500h without any sign of deactivation under a very high feedstock gas hourly space velocity value (108,000 $mL.g^{-1}.h^{-1}$). Apart from this, the influence of both GHSV values and H_2/CO_2 ratio on the 8wt%Ni/ZIF-67 catalyst performance was investigated to enhance the CO_2 conversion during the methanation reaction. Interestingly, 82% CO_2 conversion with 98% product selectivity was achieved at a reaction condition of 30,000 $mL.g^{-1}.h^{-1}$ with a feed gas (H_2/CO_2) ratio of 7:1 at 300 °C. Hence, combining porous hybrid materials such as ZIF-67 and catalytically active Ni could be a potential catalyst system for methanation reactions. Besides, the 8wt%Ni/ZIF-67 catalyst's excellent catalytic activity and profound stability under high feedstock GHSV value have widened the prospect for industrial application.

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

PEMBANGUNAN RANGKA KERJA ZEOLITIC IMIDAZOLE NICKLE-DOPED YANG SANGAT CEKAP (ZIF-67) BERASASKAN PEMANGKIN UNTUK TINDAK BALAS METANASI

Oleh

ALDOGHACHI ALI FARIS ABDULRIDHA

Mei 2023

Pengerusi : Sivasangar a/ l Seenivasagam, PhD
Fakulti : Sains

Penukaran CO_2 kepada gas asli sintetik melalui tindak balas metana semakin mendapat perhatian kerana potensinya dalam menyelesaikan cabaran penyimpanan tenaga jangka panjang dan mengurangkan pelepasan CO_2. Penghasilan mangkin yang sangat cekap yang boleh berfungsi secara aktif di bawah suhu tindak balas yang rendah adalah penting untuk hasil kemampanan tindak balas metana serta membolehkan penangkapan CO_2 menjadi lebih berpatutan. Penggunaan bahan kerangka logam-organik (MOF) yang sangat berliang seperti ZIF-67 sebagai sokongan pemangkin dengan penambahan Ni akan menjadi usaha yang menarik untuk mencapai penyebaran yang baik dan mengelakkan penggumpalan dopan semasa tindak balas metana. Oleh itu, ZIF-67 telah disintesis menggunakan 2-methylimidazole dan kobalt nitrat, dan pepejal ZIF-67 yang diperoleh telah diaktifkan di bawah ketuhar vakum pada 80 °C selama semalaman. Dopan nikel telah dicampurkan ke dalam ZIF-67 dalam julat 1-12 wt% melalui kaedah impregnasi basah, dan mangkin dikalsinkan pada 350 °C selama 4 jam di bawah aliran N_2. mangkin yang terhasil telah dicirikan menggunakan Brunauer-Emmett-Teller (BET), X-ray diffraction (XRD), Thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), Temperature-programmed desorption daripada karbon dioksida (TPD-CO_2), Temperature-programmed reduction (TPR), and High-resolution transmission electron microscopy (HRTEM). Sebelum tindak balas metana berlaku, 0.05g mangkin telah dimuatkan ke dalam reaktor tiub tetap dan pengurangan secara 'in-situ' di bawah aliran 5%H_2/seimbang Ar pada 350°C selama 3 jam. Setelah itu, aliran tersebut ditukar kepada gas reaktan untuk menguji mangkin pada suhu tindak balas yang telah ditetapkan. Tindak balas awal pemangkin metana telah dijalankan pada suhu 400°C, dengan kadar alir 108,000 $mL.g^{-1}.h^{-1}$ serta gas bahan suapan (H_2/CO_2) bernisbah 4:1. Pencirian fizikokimia pemangkin yang terhasil mempamerkan keluasan permukaan dan keliangan yang sangat tinggi serta keagihan dopan Ni sama rata di atas permukaan ZIF-67. Mangkin didapati aktif dalam penukaran CO_2, di mana kehadiran Ni secara signifikan mempengaruhi selektiviti produk dari tindak balas. Berdasarkan keputusan saringan pemangkin, mangkin 8wt%Ni/ZIF-67 ditemui paling aktif di kalangan pemangkin lain yang

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disediakan dalam tindak balas metana, dengan penukaran CO_2 yang sederhana sebanyak 56% diikuti oleh selektiviti 96% pada suhu tindak balas 300° C.

Tambahan pula, aktiviti pemangkin menunjukkan kestabilan yang tinggi, dengan penukaran sebanyak 53% dan selektiviti produk sebanyak 94% pada 500 jam tanpa sebarang tanda penyahaktifan di bawah nilai gas hourly space velocity bahan suapan yang sangat tinggi (108,000 $mL.g^{-1}.h^{-1}$). Selain itu, pengaruh kedua-dua nilai GHSV dan nisbah H_2/ CO_2 ke atas prestasi mangkin 8wt%Ni/ZIF-67 telah disiasat untuk meningkatkan lagi penukaran CO_2 semasa tindak balas metana. Hasil dapatan yang menarik dapat dilihat di mana penukaran CO_2 sebanyak 82% dengan selektiviti produk 98% tercapai pada keadaan tindak balas 30,000 $mL.g^{-1}.h^{-1}$ dengan nisbah gas suapan (H_2/ CO_2) 7:1 pada 300 °C. Oleh itu, penggabungan bahan hibrid berliang seperti ZIF-67 dan keaktifan Ni yang tinggi berpotensi menjadi sistem pemangkin yang sesuai untuk tindak balas metana. Di samping itu, aktiviti pemangkin yang cemerlang serta kestabilan yang mendalam di bawah nilai GHSV bahan suapan bagi pemangkin 8wt%Ni/ZIF-67 telah meluaskan prospek untuk aplikasi industri.

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Sivasangar a/l Seenivasagam, PhD
Senior Lecturer ChM
Faculty of Humanities, Management and Science
Universiti Putra Malaysia
(Chairman)

Taufiq Yap Yun Hin, PhD
Professor Datuk. ChM. Ts
Faculty of Science
Universiti Putra Malaysia
(Member)

Mohd Izham bin Saiman, PhD
Senior Lecturer ChM
Faculty of Science
Universiti Putra Malaysia
(Member)

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 14 March 2024

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LIST OF ABBREVIATIONS

BET	Brunauer-Emmett-Teller
CCU	Carbon Capture and Utilization
EDX	Energy Dispersive X-ray
FTIR	Fourier Transform Infrared Spectroscopy
GC-TCD	Gas Chromatography-Thermal conductivity detector
GHSV	Gas Hourly Space Velocity
HR-TEM	High-Resolution Transmission Electron Microscopy
IPCC	Intergovernmental Panel on Climate Change
MOF	Metal-Organic Framework
PCP	Porous Coordination Polymer
SBU	Secondary Building Unit
TGA	Thermogravimetric Analysis
TPD	Temperature Programmed Desorption
TPR	Temperature Programmed Reduction
UIO-66	Universitetet I Oslo-66
XPS	X-ray Photoelectron Spectroscopy
XRD	X-Ray Diffraction
ZIF-67	Zeolitic imidazolate frameworks-67

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

Global warming and climate change are two of humanity's most significant challenges. Their formation has been linked to the increase in the atmospheric concentration of greenhouse gases (Zandalinas et al. 2021). The development of human civilization and the economy's growth during the 21st century resulted in extensive use of carbon-rich fossil fuels in industrial production. Climate changes such as global warming, rising sea levels, and frequent extreme weather have occurred from the accumulation of CO_2 in the atmosphere (Fu et al. 2023). As seen in Figure 1.1, the concentration of CO_2 in the atmosphere has risen dramatically from 287 to 408 ppm since the Second Industrial Revolution (Liu et al. 2022). Therefore, the international community supports ways to reduce global CO_2 emissions to acceptable levels.

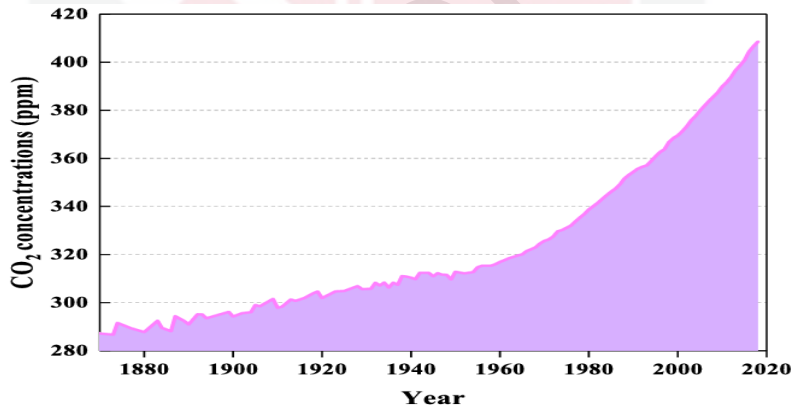

Figure 1.1: The change in atmospheric CO_2 concentrations
(Liu et al., 2022)

Several strategies have been investigated to reduce the increasing CO_2 content in the atmosphere, like converting CO_2 into products with added value. However, the CO_2 molecule's strong thermodynamic stability complicates its transformations, resulting in chemical reactions that often occur at high temperatures and pressures. Figure 1.2 summarizes the publications reporting on converting CO_2 into value-added chemicals between 2012 and 2022. Several types of research focused on using photocatalytic reactions, methane dry reforming, electrochemical reduction, and thermochemical catalytic hydrogenation to utilize CO_2. Electrocatalytic reduction of CO_2 is performed at ambient temperature and pressure. Still, it requires a significant overpotential to activate the stable CO_2 molecules (Ruiz-López et al., 2022), often resulting in an unwanted hydrogen evolution reaction (Yu et al., 2023).

On the other hand, photocatalytic CO_2 hydrogenation utilizes photoexcitation energy carriers on semiconductor photocatalysts to undergo a series of reduction and oxidation reactions (Zhang et al., 2021). However, this method generally performs less than typical thermal catalysis and has inefficient light absorption and faster recombination (Keen & Tahir, 2022). Additionally, dry reforming of methane is a reaction with potential usefulness, but it faces several challenges. These include high endothermicity, catalyst deactivation due to carbon deposition on the solid active surface, and substantial limitations imposed by equilibrium (Minardi et al., 2015).

In this context, CO_2 hydrogenation is a reaction in which carbon dioxide reacts with hydrogen to form hydrocarbons, oxygenates or other value-added chemicals with the use of a catalyst. The CO_2 hydrogenation reaction is a promising route for carbon capture and utilization (CCU) and sustainable energy storage since it converts CO_2 into value-added chemicals.

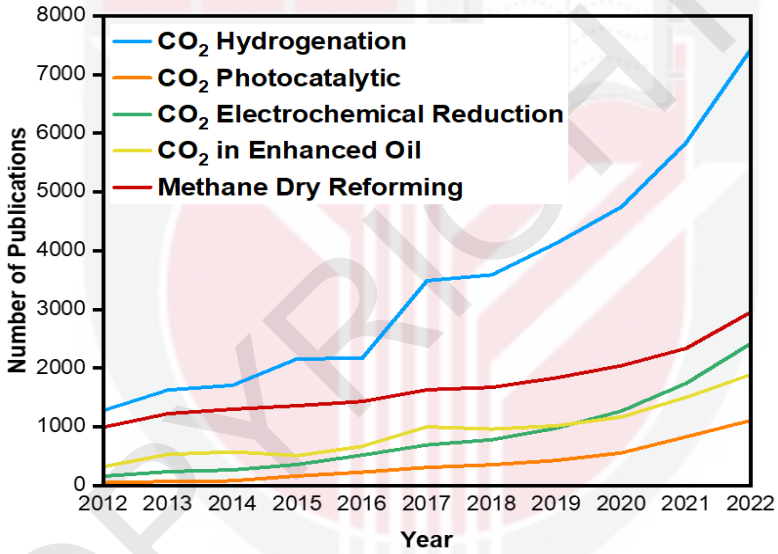

Figure 1.2: Publications on capture and utilization of carbon dioxide from 2012-2022
(Data compiled from Elsevier's database showcases the annual number of publications related to the capture and utilization of carbon dioxide)

Methanation, also called the Sabatier reaction, is a type of hydrogenation reaction, and it is one of the best-known and most-used processes. From a thermodynamic standpoint, the Sabatier reaction is an exothermic process that works most effectively at temperatures between 25 and 400 ^oC (Ashok et al., 2020). Even though the reaction is more likely to occur at low temperatures, the kinetic barrier limits its industrial applications. Thus, the hydrogenation of carbon dioxide to methane at low temperatures is only achievable with a highly effective catalyst (Fan & Tahir, 2021).

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Metal-organic frameworks (MOFs) are crystalline materials made of metal ions and organic linkers that make structures with large internal surface areas (Zurrer et al., 2020). MOFs are ideal candidates for capturing carbon dioxide and making efficient catalysts as they have ultra-high porosity and crystallinity, as well as functionalized organic linkers with distinctive characteristics such as a large surface area, high stability, and permanent porosity (Olajire, 2018). Among all MOFs, zeolitic imidazolate framework-67 is formed through the coordination interaction between cobalt metal ions and 2-methylimidazole derivatives. ZIF-67 is highly regarded due to its low-density, high surface area, precise microporosity, and excellent thermal and chemical stability. Additionally, this material is easy of preparation/regeneration, making it an attractive option for carbon dioxide separation and capture applications.

1.2 Problem statement

The carbon dioxide methanation reaction shows excellent potential for reducing carbon emissions and producing renewable fuels. However, the efficiency of the reaction is limited by the activity and stability of the catalyst. Ni-based catalysts are commonly used due to their high activity and availability. However, increasing the Ni loading on the support to enhance low-temperature activity often leads to larger Ni particles and decreased metal surface availability due to weak metal-support interaction (Ho et al., 2020). It could cause agglomeration of active catalyst sites and production of nickel carbonyl and carbon deposition, resulting in the catalyst's deactivation or an alteration in product selectivity.

Additionally, poor dispersion of active particles due to low surface area and porosity of the support leads to carbon deposition during the reaction. These issues result in decreased catalytic activity and sintering of active particles, leading to catalyst deactivation. To overcome the catalyst deactivation challenges, catalysts need to be developed with stronger interactions between the active particles and support and higher surface area and porosity for better dispersion of the active particles (Zhang and Liu 2020).

1.3 Scope of study

This work aims to synthesize a MOF-based catalyst ZIF-67 and examine the effect of metal Ni loading with varying compositions on ZIF-67. The physicochemical properties of the catalysts have been investigated using a variety of characterization analysis like X-Ray diffraction analysis, Thermogravimetric analysis, Brunauer-Emmett-Teller surface area analysis, Fourier transform infrared, High-resolution transmission electron microscopy, Temperature-programmed reduction, Temperature-programmed desorption-CO_2, and X-ray photoelectron spectroscopy. Following, the catalysts were tested for CO_2 methanation reaction in a continuous fixed-bed reactor equipped with a mass flow controller and an online GC-TCD system. Different reaction parameters were examined to achieve the best catalytic activity, such as the effect of reaction temperature, nickel loading, gas hourly space velocity (GHSV), and of reactant ratio. The catalyst with the best activity was selected for a long-term stability test to inspect if it would

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undergo deactivation or degradation over time. Therefore, the spent catalysts from the 500 hours stability test were characterized using HR-TEM and XPS to determine the appearance of coke deposition or Ni sintering.

1.4 Objectives

This project aims to produce a synthesis gas using a highly active catalyst. The objectives of this project are stated below:

1. To synthesize and characterize thermally stable various Nickel loadings added ZIF-67 based catalysts.

2. To perform catalytic CO_2 methanation reaction under a fixed bed reactor.

3. To optimize the reaction parameters (Ni loading, temperature, GHSV, and reactant ratio) of CO_2 methanation reaction.

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