

SYNTHESIS OF CERIUM OXIDE-MAGNESIUM OXIDE ADSORBENT USING
EGG-SHELL MEMBRANE BIO-TEMPLATING FOR CARBON DIOXIDE
CAPTURE

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DEDICATION

This thesis is dedicated to my parents, who taught me the real meaning of life. They will always be my number one for the rest of my life. Also dedicated to my postgraduate colleague, who always helpful for any inquiries. Finally, to my supervisor, who guided me throughout the journey as a postgraduate student in UTM.

“Learning Never Ends”

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ABSTRACT

Increased CO₂ atmospheric concentration is majorly contributed by the uncontrolled greenhouse gasses emission from rapid industrialisation. This phenomenon could lead to irreversible environmental problems such as climate change, global warming, ocean acidification and other environmental related issues. Thus, to keep this under control, several approaches have been proposed and conducted. Currently, carbon capture via metal oxide solid adsorbent adsorption is one of the approaches that is progressively studied. However, commercialised metal oxide adsorbents such as magnesium oxide (MgO) and cerium oxide (CeO₂) have several drawbacks such as poor structural and textural properties and low surface basicity, leading to low CO₂ adsorption. Therefore, the purpose of this study was to prepare mesoporous composite CeO₂-MgO (CM-BT) adsorbent via the utilisation of egg-shell membrane (ESM) as a template. The prepared adsorbents were characterised using field emission scanning electron microscopy-energy dispersion X-ray spectroscopy, transmission electron microscopy-energy dispersion X-ray spectroscopy, X-ray diffraction, nitrogen (N₂) physisorption, Fourier-transform infrared spectroscopy, thermogravimetric analysis and CO₂-temperature-programmed desorption (CO₂-TPD). The CO₂ uptake performance was evaluated at 1 atm and 300 K. It was found that mesoporous CM-BT adsorbent exhibited an enhancement in structural properties, with higher surface area (42 m²/g) and pore volume (0.185 cm³/g) compared to composite CeO₂-MgO prepared via thermal decomposition (CM-TD). CM-BT exhibited a high CO₂ uptake capacity of 5.7 mmol/g, which was 2.5-times higher than CM-TD. This was due to the increased surface basicity of CM-BT, which was associated with abundant adsorption sites of weak, medium and strong base-site. This study revealed that ESM bio-templating is a promising approach in synthesising mesoporous material adsorbent with enhanced adsorbent's physicochemical properties, resulting in increased CO₂ uptake capacity.

ABSTRAK

Perindustrian yang pesat telah menjadi penyumbang terbesar kepada pelepasan gas rumah hijau yang tidak terkawal ke atmosfera dan telah menjurus kepada peningkatan tahap kepekatan karbon dioksida di atmosfera. Fenomena ini menyebabkan berlakunya masalah kepada alam sekitar termasuklah perubahan iklim, pemanasan global, pengasidan lautan dan masalah berkaitan alam sekitar yang lain. Oleh itu, bagi memastikan perkara tersebut berapa dibawah kawalan, beberapa pendekatan telah dicadangkan dan diambil. Pada ketika ini, penjerapan karbon dioksida dengan menggunakan penjerap logam oksida adalah salah satu pendekatan yang mana sedang dikaji dengan secara progresif sekali. Walau bagaimanapun, penjerap logam oksida yang dikomersialkan seperti magnesium oksida (MgO) dan serium oksida (CeO_2) memiliki beberapa kelemahan seperti sifat struktur dan tekstur yang lemah dan kelemahan pada bes permukaan yang mana telah menjurus kepada penjerapan CO_2 yang rendah. Oleh itu, kajian ini adalah bertujuan untuk menyediakan penjerap mesopori komposit CeO_2 - MgO (CM-BT) dengan menggunakan membran kulit telur (ESM) sebagai pencontoh. Penjerap yang telah disediakan dicirikan dengan menggunakan mikroskop medan imbasan elektron penyebaran tenaga sinar-X, mikroskop penghantar elektron penyebaran tenaga sinar-X, pembelaun sinar-X, kaedah penjerapan nitrogen, spektroskopi inframerah transformasi Fourier, analisa termogravimetrik dan penyahjerapan CO_2 dengan suhu yang diprogramkan (CO_2 -TPD). Prestasi penjerapan CO_2 dinilai pada 1 atm dan 300 K. Didapati bahawa penjerap komposit CM-BT mesopori menunjukkan peningkatan sifat strukturnya dengan luas permukaan ($42\text{ m}^2/\text{g}$) dan isipadu pori ($0,185\text{ cm}^3/\text{g}$) yang lebih tinggi berbanding komposit CeO_2 - MgO yang disediakan melalui penguraian terma (CM-TD). CM-BT telah menunjukkan peningkatan kapasiti penjerapan CO_2 yang tinggi sebanyak 5.7 mmol/g, iaitu 2.5-kali lebih tinggi berbanding CM-TD. Hal ini didorong oleh kebesan permukaan penjerap CM-BT yang tinggi, yang mana berkaitan dengan bilangan tapak penjerap alkali lemah, sederhana dan kuat yang banyak. Kajian ini telah mendedahkan bahawa bio-pencontoh ESM adalah pendekatan yang meyakinkan dalam penyediaan penjerap mesopori yang memiliki sifat fizikokimia yang dipertingkatkan, yang mana menyumbang kepada peningkatan kapasiti penjerapan CO_2 .

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LIST OF SYMBOLS

Cu K α	-	Copper Potassium Alpha X radiation
dV/dlogD	-	Pore volume
P/P ₀	-	Relative pressure
wt%	-	Percentage by weight
c	-	BET Constant
n	-	Order of Reflection
λ	-	Wavelength on Incident Ray
d	-	Interplanar Spacing of the Crystal
θ	-	Incident Angle

LIST OF ABBREVIATIONS

BET	-	Brunauer-Emmett-Teller
BJH	-	Barrett-Joyner-Halenda
CCS	-	Carbon Capture & Storage
CCU	-	Carbon Capture & Utilisation
ESM	-	Egg-Shell Membrane
CeO ₂ -BT	-	Cerium Oxide-Biotemplated
MgO-BT	-	Magnesium Oxide-Biotemplated
CM-BT	-	CeO ₂ -MgO-Biotemplated
CM-x	-	CeO ₂ -MgO-Biotemplated with investigated parameter of varies pH condition
CM-y	-	CeO ₂ -MgO-Biotemplated with investigated parameter of different solvent type
CM-z	-	CeO ₂ -MgO-Biotemplated with investiagted parameter of different calcination temperature
CO ₂	-	Carbon Dioxide
FTIR	-	Fourier-Transform Infrared Spectroscopy
IPCC	-	Intergovernmental Panel on Climate Change
TEM	-	Transmission Electron Microscope
STEM	-	Scanning Transmission Electron Microscopy
HRTEM	-	High-Resolution Transmission Electron Microscopy
FESEM	-	Field Emission Scanning Electron Microscope
EDX	-	Element Dispersion X-ray Spectroscopy
SAED	-	Selected Area Electron Diffraction
TGA	-	Thermogravimetric Analysis
XRD	-	X-ray Diffraction
TPD	-	Temperature Programmed Desorption

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Rapid urbanisation and industrialisation are significant contributors to the increasing CO₂ emissions into the atmosphere (Chen et al., 2019). According to the statistical data (Richie, 2017), Asia countries are the highest contributor amounting to 19 billion tons of CO₂, led by China with 53 % of global CO₂ emission. The Intergovernmental Panel on Climate Change (IPCC) has reported that the atmospheric CO₂ concentration will reach up to 570 ppm in the year 2100. This increasing CO₂ concentration will be causing an irreversible environmental issue such as global warming due to the elevation of global average temperature (Rahimi et al., 2019) and ocean acidification (Guo et al., 2019). Ocean acidification is result from the gas exchange between air and ocean surface. Hence, the high dissolved inorganic carbon will decrease the pH of the surface ocean (Aghaie et al., 2018). Therefore, many studies have been conducted to minimise CO₂ emissions into the atmosphere such as through the carbon capture approach then further proceed with utilisation and storage process operated under CCU and CCS application, respectively.

Carbon capture can be achieved through several techniques such as absorption, cryogenic, membrane and adsorption (Azmi & Aziz, 2019). Nowadays, the most industrial implemented method in capturing CO₂ is the absorption technique. However, the currently used absorption technique faced several significant drawbacks: high equipment corrosion rate, high energy consumption during the regeneration stage, and loss of solvent in the presence of SO₂, NO₂ and O₂ and also through evaporation (Aghaie et al., 2018; Jiang et al., 2019; Wang et al., 2014; Yu et al., 2012). These disadvantages are linked with the inherent properties of amine, which have high vapour pressure, corrosive nature, and high energy input for regeneration (Aghaie et

al., 2018). Therefore, due to these limitations, the adsorption method seems to be much promising method due to the advantage adsorption process such as easy handling and recovery process, low CO₂ separation cost, low regeneration energy requirement and economical solid adsorbent (Jiang et al., 2019).

Metal oxide-based adsorbent such as magnesium oxide (MgO) possesses a promising adsorbent characteristic which beneficial in capturing CO₂, such as its high theoretical uptake capacity (24.8 mmol/g), low toxicity, tuneability porous structure, having appropriate surface basicity strength, and wide range of adsorption temperature up-to 673 K (Azmi & Aziz, 2019; Hu et al., 2019). However, conventional MgO tends to suffer an issue such as limited adsorption capacity and low adsorption rate (Li et al., 2020). This issue could be related to poor physicochemical properties (low basicity strength, surface area and pore volume), limiting the binding of the CO₂ to the adsorbent active binding site. Therefore, it is desirable to fabricate an adsorbent with excellent physicochemical properties. This tuning approach could be achieved by composite MgO with other metal oxide/non-metal and choosing a suitable synthesis method.

In addition, composite MgO adsorbent has also attracted much attention, which composite MgO adsorbent offers better physicochemical properties and surface basicity strength, leading to high CO₂ uptake capacity (Chen et al., 2019; Yu et al., 2018). According to (Liu et al., 2015), this reactivity is correlated with the synergetic effect between compositing metal (e.g. CeO₂) and other doped metal (M) such as MgO. This effect facilitates the ion exchange between Mⁿ⁺ / Mⁿ⁺¹ and Ce³⁺ and Ce⁴⁺, which act as an active site for trapping CO₂. In addition, as reported by (Yu et al., 2018), the MgO-based adsorbent was successfully prepared via urea co-precipitation. It is found that composite Al₂O₃-MgO and CeO₂-MgO have exhibited better surface strength basicity with the evidence of CO₂-TPD than pure MgO. Although, every composite MgO-based adsorbent also possessing a varies surface basicity strength. In addition, decent adsorbent's physicochemical properties also reported influenced by the type of synthesis method used. Each synthesis method will result in different adsorbent's textural and structural properties that affecting its adsorption performance (Guo et al., 2020). These structural enhancements were usually found related to surfactant

templates utilisation, such as Cetyltrimethylammonium Bromide (CTAB). However, this surfactant template possesses several drawbacks, such as the formation of aggregated adsorbent nanocrystals with a disordered mesoporosity due to the relatively weak interaction between surfactant and the adsorbent-based species (Yang et al., 2017), costly (Wang et al., 2009) and complicated procedure (Abarna et al., 2016; Li et al., 2015).

Consider minimising the issue faced by the current surfactant-used synthesis method, the bio-templating method is one of the alternative approaches. The Bio-templating method uses a natural-based material (biomaterial) as a template (Ma et al., 2019). Biomaterial used will act as a pore generator for adsorbent where metal ions will be loaded to and then removed via the calcination process. The remaining metal-oxide residue will be formed as the parental bio-template structure used (He et al., 2019). For instances, (Witoon et al., 2014) has reported on the chitosan bio-templated Ca-based adsorbent toward CO₂ capture application. It is found that the chitosan bio-templated Ca-based adsorbent's morphological feature was influenced by the ratio of the chitosan used and has resulted to higher CO₂ capture than pure CaO and surfactant-used CaO. Considering the high fibrous structure material that could be induced to the increase of adsorbent's surface area, eggshell membrane (ESM) is a potential candidate to be utilised as a bio-templating material. ESM possess a 3D interwoven natural protein fibrous network structure (Chen et al., 2019; Preda et al., 2020), leading to the generation of mesoporous structured adsorbent (Fan et al., 2016; He & Yang, 2018).

In this study, mesoporous composite CeO₂-MgO was synthesised using the bio-templating method with ESM as a template. As a comparison, composite CeO₂-MgO adsorbent was also synthesised via the thermal decomposition method. The prepared adsorbents were all subjected to characterisation by using X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy, Nitrogen (N₂) adsorption-desorption, High-resolution transmission electron microscopy (HRTEM), Field

emission scanning electron microscopes (FESEM), Energy-dispersive X-ray (EDX) spectroscopy, Thermogravimetric analysis (TGA) and Temperature-programmed desorption of carbon dioxide (CO₂-TPD). The adsorbent's CO₂ uptake capacity was tested under CO₂ gas conditions at atmospheric pressure and temperature.

1.2 Problem Statement

Commercial MgO adsorbent seems to suffer several drawbacks, such as low specific surface area and low surface basicity. This issue has caused low CO₂ uptake capacity since the accessibility of the CO₂ molecule to the adsorbent's active site and number of basic attachment site are very limited. Thus, enhancement approaches are needed. MgO's physicochemical properties can be tuned by compositing with other metal oxide and fabricating a high surface area MgO adsorbent. Metal oxide composite (e.g. with CeO₂) can enhance the adsorbent's physicochemical properties by tuning the adsorbent's surface basicity strength. Then, influencing adsorbent CO₂ uptake capacity. Utilising a proper synthesis method, such as a surfactant-template-based method, could also improve MgO's physicochemical properties. However, a common surfactant used, such as CTAB utilised is costly. Hence, using biomass as a template is an alternative to minimising this issue since the bio-material template is abundantly available. Therefore, it is expected that using ESM as a template in the preparation phase will result in the generation of high surface area adsorbent, which further contributes to the enhancement of the adsorbent's CO₂ uptake capacity.

1.3 Objectives of the Study

The objectives of this study are;

1. To synthesise mesoporous composite CeO₂-MgO by using the thermal decomposition method and ESM bio-templating method.

2. To characterise the prepared adsorbent using X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy, Thermogravimetric analysis (TGA), N₂ adsorption-desorption isotherm, Temperature-programmed desorption (TPD), Field emission scanning electron microscope (FESEM), High-resolution transmission electron microscopy (HRTEM) and Energy-dispersive x-ray (EDX) spectroscopy.
3. To test the prepared adsorbent on the CO₂ adsorption uptake capacity.

1.4 Scope of the Study

- a) The composite CeO₂-MgO adsorbent was synthesised via the thermal decomposition and eggshell membrane (ESM) templating methods. Initially, the adsorbent was prepared by using thermal decomposition method with different adsorbent CeO₂-MgO molar ratio of (0:1), (0.25:0.75), (0.5:0.5), (0.75:0.25) and (1:0). This to determine the optimum CeO₂-MgO molar ratio that yields high CO₂ uptake capacity. Then, single metal and composite adsorbents with high CO₂ uptake capacity were further investigated on the ESM bio-templating method's effect. Moreover, to further understand the ESM bio-templating method, several synthesis parameters such as the solution's pH condition, type of solvent used, and different calcination temperature was investigated on the composite CeO₂-MgO adsorbent.
- b) The physicochemical properties of the prepared adsorbents were investigated by using several characterisation analyses, as listed in Section 1.3 (Objective). The morphological features of the prepared adsorbent were examined and observed using FESEM and HRTEM analysis with EDX elemental mapping. Moreover, the X-ray diffraction analysis was executed to investigate the crystallinity properties of all prepared samples. For the analysis using FTIR spectroscopy, the adsorbents surface functional group was examined. The structural properties such as surface area and pore volume were investigated

via N₂ adsorption-desorption isotherm. The TGA was conducted to study the thermal decomposition behaviour of the prepared adsorbent. Lastly, the adsorbent surface basicity strength was evaluated using TPD with CO₂ gas as a probe.

- c) The CO₂ adsorption process was conducted for all prepared adsorbents using a fixed bed U-shaped adsorption column equipped with a CO₂ analyser instrument with 100 % CO₂ flow condition under ambient conditions with 5 adsorption-desorption cycles.

1.5 Significant of Study

In this study, the mesoporous composite CeO₂-MgO adsorbent was synthesised using the bio-templating method with the ESM utilisation as a template and expected to exhibit a high CO₂ uptake capacity. This high CO₂ uptake capacity was induced by the enhanced adsorbent's surface area and surface basicity strength/defect site generated by ESM bio-templating and composite CeO₂-MgO, respectively. An abundance of porous surfaces provides much efficient gas diffusion into the adsorbent's interior structure, and further increases active binding site exposure. In addition, this bio-template is a promising template to be used since it possesses a 3-dimensional structure contributing to the generation of mesoporous structure adsorbent. Moreover, it appears to be more economical due to abundantly available bio-material sources. These improvements and approaches were beneficial to the adsorption process in a wide range of applications such as post-combustion, gas purification, bio-gas purification, etc.

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Journal with Impact Factor

1. **A.H.Ruhaimi**, M.A.A. Aziz, A.A. Jalil. (2021). Magnesium oxide-based adsorbent for carbon dioxide capture: Current progress and future opportunities. *Journal of CO₂ Utilisation*, 43, 101357. <https://doi.org/10.1016/j.jcou.2020.101357> (**Q1, IF:7.132**)
2. **A.H. Ruhaimi** and M.A.A. Aziz, (2021). High-performance flake-like mesoporous magnesium oxide prepared by eggshell membrane template for carbon dioxide capture. *Journal of Solid State Chemistry*, 300, 122242. (**Q2, IF:2.726**)
3. **A.H. Ruhaimi** and M.A.A. Aziz, (2021). Spherical CeO₂ nanoparticles prepared using an egg-shell membrane as a bio-template for high CO₂ adsorption. *Chemical Physics Letters*, 779, 138842. (**Q2, IF:2.328**)

Indexed Journal

4. **Amirul Hafiz Ruhaimi**, Haziq Fikri Zaini and Muhammad Arif Ab Aziz. (2020). Effect of ceria/surfactant molar ratios on the formation of mesoporous ceria nanoparticles and its application in CO₂ capture. *Malaysian Journal of Catalysis*, 4, 44-48. (**Indexed by ROAD**)
5. **A.H. Ruhaimi**, C.C. Teh, M.A.A. Aziz, (2021). Mesoporous Magnesium Oxide Adsorbent Prepared via Lime (*Citrus aurantifolia*) Peel Bio-templating for CO₂ Capture. *Bulletin of Chemical Reaction Engineering & Catalysis*. 16(2), 36673. (**Indexed by Scopus, Q3**)