SYNTHESIS AND CHARACTERIZATION OF AMINE-IMPREGNATED MESOPOROUS CERIA NANOPARTICLES FOR CARBON DIOXIDE CAPTURE

AHMAD AIMAN BIN AZMI

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School of Chemical and Energy Engineering
Faculty of Engineering
Universiti Teknologi Malaysia

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DEDICATION

This thesis is dedicated to my parents, who taught me the real meaning of perseverance. They always be my number one for the rest of my life. Also dedicated to my friend, Din who always have been my go-to person for any inquiries. Finally, to my supervisor who guided me throughout the journey as a postgraduate student in UTM.

“Greatness, from small beginnings”

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ABSTRACT

Carbon dioxide (CO_2) contributes more than 60% towards global warming especially from the fossil-fuel burning activity. Hence, technologies such as carbon capture storage and utilization was introduced. Among the carbon capture technologies, adsorption by porous materials have been used as adsorbent material. Ceria has been chosen in this study. Commercial ceria has several drawbacks such as less porosity and surface area which reduce the availability of CO_2 adsorption site. The objective of this study is to prepare a mesoporous ceria nanoparticles (MCN) via hydrothermal and sol-gel methods. 3-aminopropyltrimethoxysilane (APTMS) was used in order to improve the CO_2 capture performance. The characterization of all samples were carried out by nitrogen adsorption-desorption isotherm, X-ray diffraction, transmission electron microscopy, Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis, pyrrole and CO_2 adsorbed FTIR spectroscopy. The performance of the samples were tested by CO_2 adsorption at pressure range between 6-900 mmHg and 298 K. The preparation parameters were determined via hydrothermal method at calcination temperature of 673 K, pH 9 and ceria/surfactant ratio 2. These parameters were then applied to sol-gel method and the prepared mesoporous adsorbent produced high surface area (76.0 m^2 g^{-1}), large pore size (0.100 cm^3 g^{-1}), large pore volume (5.3 nm) and high CO_2 uptake (213.8 µmol g^{-1}). The adsorbent also shows high thermal stability because it can retain up to 1171 K. The proposed CO_2 adsorption mechanism was elucidated from the CO_2 adsorbed FTIR spectroscopy. For MCN-2S, CO_2 was adsorbed onto oxygen basic, oxygen vacancy and hydroxyl site on MCN which formed monodentate, bidentate, polydentate and hydrogen carbonate. In addition to these carbonate species, the adsorption of CO_2 on APTMS/MCN-2S also occurred through the formation of carbamate species. Low CO_2 adsorbed on the APTMS/MCN-2S might be due to the utilization of available oxygen basic sites by APTMS molecules. This study exhibited that MCN adsorbent prepared by sol-gel method showed a potential to be applied at industrial scale due to the rapid preparation method, high thermal stability and high CO_2 uptake capacity.

ABSTRAK

Karbon dioksida (CO_2) menyumbang melebihi 60% terhadap pemanasan global daripada yang berlaku terutama daripada aktiviti pembakaran bahan api fosil. Oleh itu, teknologi seperti penangkapan dan penyimpanan karbon telah diperkenalkan. Di antara kesemua teknologi penangkapan karbon, teknik penjerapan oleh bahan berliang telah digunakan sebagai bahan penjerap. Ceria telah dipilih di dalam kajian ini. Ceria komersial mempunyai beberapa kekurangan seperti kurangnya liang dan luas permukaan yang rendah mengakibatkan pengurangan tapak penjerapan CO_2. Objektif kajian ini dilakukan untuk menyediakan nanopartikel ceria berliang bersaiz meso menggunakan kaedah hidroterma dan sol-gel. 3-aminopropiltrimetoxisilana (APTMS) juga digunakan untuk menambahbaik kesan ke atas penangkapan CO_2. Semua bahan yang telah disintesis dicirikan menggunakan kaedah penjerapan nitrogen, pembelauan sinar-X, mikroskop penghantaran elektron, spektroskopi inframerah transformasi Fourier (FTIR), analisa termogravimetrik, spektroskopi FTIR terjerap pirol dan CO_2. Prestasi sampel diuji menggunakan kaedah penjerapan CO_2 pada julat tekanan 6-900 mmHg dan 298 K. Parameter ditentukan melalui kaedah hidroterma pada suhu pengkalsian 673 K, pH 9 dan ceria/surfaktan bernisbah 2. Kemudian, parameter tersebut diguna pakai untuk kaedah sol-gel dan menghasilkan bahan penjerap yang mempunyai luas permukaan yang tinggi (76.0 m^2 g^{-1}), liang bersaiz besar (0.100 cm^3 g^{-1}), isipadu liang yang besar (5.3 nm) dan penjerapan CO_2 yang tinggi (203.9 μmol g^{-1}). Bahan penjerap ini menunjukkan kestabilan terma yang tinggi kerana mampu kekal sehingga suhu setinggi 1171 K. Mekanisma penjerapan CO_2 dijelaskan melalui spektroskopi FTIR terjerap CO_2. Bagi MCN-2S, CO_2 terjerap pada oksigen asas, kekosongan oksigen dan tapak hidroksil yang membentuk monodentat, bidentate, polidentat dan hidrogen karbonat. Tambahan dari spesies karbonat ini, penjerapan CO_2 pada APTMS/MCN-2S juga berlaku melalui pembentukan spesies karbamat. Penjerapan CO_2 yang rendah pada APTMS/MCN-2S disebabkan oleh pengunaan permukaan oksigen asas oleh molekul APTMS. Kajian ini membuktikan penjerap MCN yang disediakan melalui kaedah sol-gel mempunyai potensi untuk diaplikasikan dalam skala industri disebabkan beberapa faktor seperti prosedur yang cepat, kestabilan terma yang tinggi dan penjerapan CO_2 yang tinggi.

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LIST OF ABBREVIATIONS

APTMS	-	3-Aminopropyltrimethoxysilane
BET	-	Brunauer-Emmett-Teller
BJH	-	Barrett-Joyner-Halenda
CCS	-	Carbon Capture & Storage
CeO_2	-	Ceria Oxide/Cerium Oxide
CO_2	-	Carbon Dioxide
CTAB	-	Cetyl Trimethylammonium Bromide/Cetrimonium Bromide
FTIR	-	Fourier-Transform Infrared Spectroscopy
IR	-	Infrared Radiation
KBr	-	Potassium Bromide
MCN	-	Mesoporous Ceria Nanoparticles
N_2	-	Nitrogen
OSC	-	Oxygen Storage Capacity
SEM	-	Scanning Electron Microscope
STP	-	Standard Conditions for Temperature And Pressure
TEM	-	Transmission Electron Microscope
TGA	-	Thermogravimetric Analysis
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

a	-	Weight Percent (%)
b	-	Adsorption at 2 Bar
Cu Kα	-	Copper Potassium Alpha X Radiation
P/P_0	-	Relative Pressure
wt%	-	Percentage By Weight
v	-	Volume of Adsorbed Gas
v_m	-	Monolayer Adsorbed Volume
p	-	Equilibrium Gas Pressure
p_0	-	Saturation Pressure
c	-	BET Constant
n	-	Order Of Reflection
λ	-	Wavelength On Incident Ray,
d	-	Interplanar Spacing Of The Crystal
θ	-	Incidence Angle

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Since the dawn of industrialization era, CO_2 contributes greater than 60% to global warming due of its enormous emission to the atmosphere (Albo *et al.*, 2010). Environment and economy across continents are left unprotected due to the harmful CO_2 levels. A report shown by Intergovernmental Panel on Climate Change (IPCC) stated a possible rise from 274.1 K to 279.4 K globally before 2100 (Saboori *et al.*, 2012). As an awareness, many countries and organizations have taken part in establishing global regulations of CO_2 emissions. For instance, in the United Climate Change Conference that was held in city of Paris in December 2015, new objective was set to ensure the global temperature increase be limited to 275 K (Ojeda *et al.*, 2017). In order to reduce the CO_2 emission, International Energy Agency (IEA) recommended to fully utilize and develop an advanced technology known as capture and storage (CCS). CCS is the potential solution to mitigate the anthropogenic CO_2 emissions from industrialization, hence the urgency to develop this technology is paramount.

The general concept of CCS is to trap CO_2 before it penetrates the atmosphere and store it in safe storage for future use. CCS involves many processes for CO_2 such as capture, separation, storage, transportation and monitoring. In industrial scale, there are numerous typical methods implemented such as absorption, adsorption, membrane separation and cryogenics treatment (Leung *et al.*, 2014). However since 1930s, absorption process using amines solution is the most efficient one currently used in industry (Wang *et al.*, 2011a). Unfortunately, the absorption has multiple limitations for instance low absorption measurements, expensive recycling cost, high corrosiveness, inferior stability, solvent loss and high viscosity challenges (Liang *et al.*, 2016). One of the solution is to synthesize a porous adsorbents consists of excellent textural properties including high surface area, big pore volume and outstanding pore size distribution (Sreenivasulu *et al.*, 2015).

The adsorption process is relatively important due to the reversible and enhanced efficiency by altering the structure of the adsorbent materials. Nowadays, there are many studied porous supports used in adsorption such as activated carbon, zeolites, mesoporous silica, metal oxide framework includes metal–organic-frameworks (MOFs) and covalent–organic-frameworks (COFs) (Coromina *et al.*, 2016; Fisher and Gray, 2015; Nguyen *et al.*, 2016; Verdegaal *et al.*, 2016). Latterly, mesoporous based adsorbent has been thoroughly investigated by many researchers displayed in Figure 1.1 below. High surface area, easy modification of pore structure, good stability, attractive surface modification, excellent regeneration ability and smooth movement of reactant are the reasons to choose mesoporous adsorbent (Ahmed *et al.*, 2016).

Figure 1.1 Database regarding progress of publications against years in 11 years duration extracted from directory of ISI Web of Science by key in the phrase "mesoporous CO_2 capture"

However, the main hindrance of this porous adsorbent can be attributed with their neutrality charges of surface that results in the restraint of maximum performance during CO_2 capture. The incorporation of highly interacted amine groups on support surface is to improve its polarization and boost CO_2 uptake capacity (Nugent *et al.*, 2013). Such adsorbents will have numerous advantages like eliminating corrosion problems, lowering the energy usage for regeneration purposes and reducing amine suitable synthesis route, shortest duration for complete preparation and optimum parameters which produce effective mesoporous ceria adsorbent.

The report on CO_2 adsorption mechanism with mesoporous ceria support is scarce. This is because the study on ceria oxide in CO_2 capture application is limited compared with others support such as silica, zeolites and metal organic framework (MOFs). Low CO_2 uptake can be overcome by functionalization with amine groups on the support surface. The introduction of amine species will stabilize and increase the adsorption rate due to their high CO_2 affinities Moreover, reports on ceria functionalized with any amine group such as APTMS is scarcely reported. A proposed mechanism about CO_2 adsorption on both pristine mesoporous ceria and mesoporous ceria functionalized with APTMS was studied for comprehensive understanding.

1.3 Objectives of Study

The objectives of this study are;

(a) To synthesis mesoporous ceria nanoparticles (MCN) by hydrothermal method and sol-gel method and loaded APTMS on the MCN.

(b) To study the physicochemical properties of the synthesized MCN and APTMS/MCN adsorbents.

(c) To examine the performance of the adsorbents on CO_2 adsorption.

(d) To investigate the mechanism of CO_2 adsorption over the adsorbents.

1.4 Scope of Study

(a) The MCNs were synthesized via hydrothermal method and sol-gel method. In the hydrothermal method, the preparations were separated into three different parameters which were calcination temperature (673, 723 K, 773 K, 823 K), pH value (1, 5, 7, 9) and ceria to surfactant ratio (0.5, 1.0, 1.5, 2.0). While in sol-gel method, the parameters were set to calcination temperature of 673 K, pH value 9 and ceria/surfactant/ratio 2.0. The liquid amine used to functionalize with the MCN was APTMS via wet impregnation method. The amount of amine loaded onto 4 g of MCN support was 10 wt%.

(b) The characterizations were carried out by using XRD, TEM, N_2 adsorption-desorption isotherm, FTIR spectroscopy, pyrrole and CO_2 adsorbed FTIR spectroscopy and TGA. Powder X-ray diffraction studies were executed via Cu Kα radiation, $2\theta = 2 - 90°$ and the analysed data was evaluated at room temperature. While the TEM analysis was performed at an accelerating voltage of 200 kV. For the N_2 adsorption desorption isotherm analysis, the weights of all samples were weighed 0.05 g. The specific surface area and the mesopore size distributions of the samples were investigated using Brunauer–Emmett–Teller (BET) method and Barrett–Joyner–Halenda (BJH) method, respectively. The pore size distribution and pore volume were analysed from desorption isotherms. For FTIR spectroscopy analysis, the samples were scanned in the range of mid infrared region (400 cm^{-1} - 4000 cm^{-1}). For TGA analysis, the heating process of the adsorbents were set at the scanning rate of 10 K min^{-1} from 300 K to 1200 K under the flow of nitrogen stream.

(c) The CO_2 adsorption measurement was recorded at pressure range between 6-900 mmHg and 298 K.

(d) The mechanistic study was investigated via in situ CO_2 adsorption by FTIR spectroscopy.

1.5 Significant of Study

In this study, MCN prepared by sol-gel method functionalized with APTMS as an adsorbent for CO_2 capture was synthesized. The synthesis of MCN by sol-gel method is more economical because it is rapid and simple. Furthermore, this synthesis route utilized lower temperature during adsorbent preparation. In terms of efficiency, MCN prepared by sol-gel method has shown high surface area and high CO_2 uptake of 76.0 $m^2\ g^{-1}$ and 213.8 $\mu mol\ g^{-1}$, respectively. Special features prepared by sol-gel method is that the MCN adsorbent exhibited unimodal pore distribution. This properties resulted in the high CO_2 uptake up to six times larger compared to the previous study reported by Yoshikawa *et al.* (2014) which exhibited a CO_2 uptake up of 30.0 $\mu mol\ g^{-1}$ for prepared mesoporous CeO_2. A plausible mechanism for CO_2 adsorption on pristine MCN and MCN functionalized with APTMS was proposed. CO_2 adsorbed on MCN produced hydrogen carbonate, monodentate carbonate, bidentate carbonate and polydentate carbonate. This can be related to the richness of significant active sites such as oxygen basic sites and oxygen vacancy which has potential to store more oxygen and further increase the CO_2 adsorption. However, functionalization of APTMS on MCN has shown a negative effect might be due to the inclusion of APTMS structure on the MCN framework which leads to a low formation of carbonate species.

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