

**SYNTHESIS AND CHARACTERISATION OF CARBON
AEROGEL DERIVED FROM CARBOXYMETHYL
CELLULOSE AS A HYDROGEN STORAGE MATERIAL**

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Thesis submitted in partial fulfilment for the degree of
MASTER OF SCIENCE

UNIVERSITI SAINS ISLAM MALAYSIA

September 2023

AUTHOR DECLARATION

I hereby declare that the work in this thesis proposal is my own unless specified and duly acknowledged by quotation.

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ACKNOWLEDGEMENTS

All praises to Allah and His blessing for the completion of this thesis. I thank Allah for all the opportunities, trials, and strength that have been showered on me to finish writing the thesis. I experienced so much during this process, not only from the academic aspect but also from the aspect of personality. My humble gratitude to the holy Prophet Muhammad (Peace be upon him) whose way of life has been a continuous guide for me.

First and foremost, I would like to sincerely thank my main supervisor, Dr Syaza Azhari, for her guidance, understanding, patience and most importantly, for providing positive encouragement and a warm spirit for me to finish this thesis. It has been a great pleasure and honour to have her as my supervisor. I also want to thank my co-supervisor, Dr Nur Atiqah Nasir for her guidance and help in making sure my thesis progress is on track. I am glad to have her as my co-supervisor. Besides, I would like to express my heartfelt gratitude to Dr Muhammad Zamir Othman, my previous supervisor for this thesis, for giving me the opportunity and trust to further my study.

My deepest gratitude goes to all my family members. It would not be possible to write this thesis without the support from them. I would like to thank my dearest father Ab Sabar Abdul Rahman, my mother Fatimah Ismail, my sister Fasihah, my youngest brother Ahmad Sauffi Al-Buqhari, my brother-in-law Ahmad Shafari, and my niece; Nur Asheeqa Safiyya and Nur Asheeqa Saqeena.

I would sincerely like to thank my laboratory partner from foundation until master, Syawal Aiman. I also want to extend my thanks to the Laboratory Staff in the Faculty of Science and Technology, Universiti Sains Islam Malaysia (USIM), especially Mr Syamsul Kamar, Mr Muhamad Izuan and Mr Muhammad Asyraf for their help and support in laboratory works. Finally, I want to thank wholeheartedly the management of Universiti Sains Islam Malaysia.

May Allah shower the above cited personalities with success and honour in their life.

ABSTRAK

Pendekatan langsung, mudah dan kos rendah untuk mensintesis komposit aerogel karbon (CA) telah ditunjukkan dalam penyelidikan ini melalui pengkarbonan aerogel natrium karboksimetil selulosa melalui sol-gel dan proses pengeringan beku. Selain daripada ion magnesium digunakan sebagai penambah untuk CA, terdapat beberapa ion logam lain termasuk mangan, nikel, dan zink dalam langkah penyediaan. Ion magnesium menunjukkan ciri terbaik penambah untuk CA kerana ia kekal semasa pengkarbonan pada kepekatan yang berbeza. Struktur dan morfologi komposit karbon aerogel-magnesium (CA-Mg) dicirikan menggunakan mikroskop pengimbasan pelepasan medan, spektroskopi inframerah transformasi Fourier (FTIR), pembelauan sinar-X (XRD), dan teknik Brunauer-Emmett-Teller (BET). Keupayaan komposit CA-Mg untuk bertindak sebagai bahan simpanan hidrogen dianalisis menggunakan analisis penyahserapan berprogram suhu. Spektrum FTIR bagi komposit CA-Mg menunjukkan pengkarbonan yang lengkap, kerana kurang puncak diperhatikan berbanding dengan CA tulen, kerana kehadiran Mg^{2+} menjadi faktor utama dalam pengkarbonan yang lengkap. Analisis XRD komposit CA-Mg menunjukkan puncak pembelauan MgO (Periclase) yang menunjukkan penjaan MgO semasa proses pengkarbonan komposit CA-Mg dan menjadi punca hilangnya puncak OH dalam spektrum FTIR yang bermaksud, $MgCl_2$ telah terurai sepenuhnya menjadi MgO. Oleh itu, komposit mempamerkan ciri-ciri CA dan MgO. Komposit CA-Mg terdiri daripada struktur berliang dengan luas permukaan spesifik yang tinggi iaitu $101.4407 \text{ m}^2/\text{g}$ dan $0.002 \text{ mol Mg}^{2+}$ ialah kepekatan optimum untuk mensintesis komposit CA-Mg. Sebagai calon berpotensi untuk bahan simpanan hidrogen, komposit CA-Mg menunjukkan suhu penyahhidrogenan awal pada $377.22 \text{ }^\circ\text{C}$ di mana mereka menyahserap jumlah maksimum gas hidrogen pada 0.168% . Kajian ini menekankan potensi untuk menggunakan CA sebagai bahan simpanan hidrogen, yang memenuhi matlamat ketujuh Matlamat Pembangunan Lestari (SDG): Tenaga yang berpatutan dan bersih, serta matlamat Jabatan Tenaga (DOE) Amerika Syarikat menggunakan bahan berasaskan karbon.

Kata kunci: aerogel karbon, karboksimetil selulosa, pengkarbonan, storan hidrogen

ABSTRACT

A direct, simple, and low-cost approach in synthesising carbon aerogel (CA) composites has been demonstrated in this research through the carbonisation of sodium carboxymethyl cellulose aerogels via sol-gel and freeze-drying processes. Magnesium ions are used as an enhancer for CA among several metal ions including manganese, nickel, and zinc in the preparation step. Magnesium ions shows the best characteristic of CA enhancer as it does not lose during carbonisation at different concentration. The structure and morphology of carbon aerogel-magnesium (CA-Mg) composites are characterised using field emission scanning electron microscopy (FESEM), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and Brunauer-Emmett-Teller (BET) techniques. The ability of CA-Mg composites to act as a hydrogen storage material is analysed using temperature programmed desorption analysis. The FTIR spectra of CA-Mg composites show the completion of carbonisation, because less peak is observed compared to pure CA, as the presence of Mg^{2+} becomes the main factor in the completion of carbonisation. XRD analysis of CA-Mg composites shows the diffraction peaks of MgO (Periclase) which indicate the generation of MgO during carbonisation process of CA-Mg composites and become the reason for the disappearing of OH peak in the FTIR spectra which means, $MgCl_2$ has been fully decomposed into MgO. Thus, the composites exhibit the characteristic features of CA and MgO. The CA-Mg composites are made up of porous structures with a high specific surface area of $101.4407 \text{ m}^2/\text{g}$ and 0.002 mol of Mg^{2+} is the optimum concentration for synthesising CA-Mg composites. As a potential candidate for a hydrogen storage material, the CA-Mg composites showed an initial dehydrogenation temperature of $377.22 \text{ }^\circ\text{C}$ where they desorbed the maximum amount of hydrogen gas at 0.168% . This study emphasises the potential for using CA as a hydrogen storage material, which fulfils the seventh goal of the Sustainable Development Goals (SDG): Affordable and clean energy, as well as Department of Energy (DOE)'s goal of using carbon-based materials.

Keywords: carbon aerogel, carboxymethyl cellulose, carbonisation, hydrogen storage

الملخص

تم توضيح طريقة مباشرة وبسيطة ومنخفضة التكلفة لتركيبة مركبات الهلام الهوائي الكربوني (CA) في هذا البحث من خلال كربنة أيروجيل كربوكسي ميثيل السليلوز الصوديوم عبر هلام محلول سولار وعمليات التجفيف بالتجميد. تستخدم أيونات المغنيسيوم كمحسن لـ CA بين العديد من أيونات المعادن بما في ذلك المنغنيز والنيكل والزنك في خطوة التحضير. تُظهر أيونات المغنيسيوم أفضل خصائص مُحسّن CA لأنها لا تفقد أثناء الكربنة بتركيزات مختلفة. تتميز بنية وشكل مركبات الكربون الهوائي / المغنيسيوم (CA-Mg) باستخدام المجهر لمسح انبعاث المجال، والتحليل الطيفي للأشعة تحت الحمراء (FTIR)، وانحراف الأشعة السينية (XRD)، وتقنيات Brunauer-Emmett-Teller (BET). يتم تحليل قدرة مركبات CA-Mg على العمل كمواد لتخزين الهيدروجين باستخدام تحليل الامتصاص المبرمج بدرجة الحرارة. تُظهر أطياف FTIR لمركبات CA-Mg اكتمال الكربنة، لأنه لوحظ وجود ذروة أقل مقارنة بـ CA النقي، حيث يصبح وجود Mg^{2+} هو العامل الرئيسي في إكمال الكربنة. يُظهر تحليل XRD لمركبات CA-Mg قمم الهيدروكسيد لـ MgO (Periclase) والتي تشير إلى توليد MgO أثناء عملية الكربنة لمركبات CA-Mg وتصبح سبب اختفاء ذروة OH في أطياف FTIR مما يعني أن $MgCl_2$ كان يتحلل بالكامل إلى MgO. وبالتالي، فإن المركبات تظهر السمات المميزة لـ CA وMgO. تتكون مركبات CA-Mg من هياكل مسامية بمساحة سطح محددة عالية تبلغ m^2/g و 101.440 و 0.002 مول من Mg^{2+} هو التركيز الأمثل لتخليق مركبات CA-Mg. كمرشح محتمل لمادة تخزين الهيدروجين، أظهرت مركبات CA-Mg درجة حرارة مبدئية لنزع الهيدروجين تبلغ 377.22 درجة مئوية حيث تمتص أقصى كمية من غاز الهيدروجين عند 0.168%. تؤكد هذه الدراسة على إمكانية استخدام CA كمواد لتخزين الهيدروجين، والتي تحقق الهدف السابع من أهداف التنمية المستدامة (SDG): طاقة نظيفة وبأسعار معقولة، بالإضافة إلى هدف وزارة الطاقة المتمثل في استخدام المواد القائمة على الكربون.

الكلمات المفتاحية: إيروجيل الكربون، كربوكسي ميثيل السليلوز، الكربنة، تخزين الهيدروجين

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LIST OF SYMBOLS

%	Percentage
bar	Metric Unit of Pressure
Pa	Pascal
°C	Temperature (degree Celsius)
keV	kilo electronvolt
mL	milliliter
mol	mole
θ	theta
°	degree
h	Hour
kg/m ³	Unit of density (kilogram per unit cubic meter)
m ² g ⁻¹	Specific surface area (meter square per unit gram)
nm	nanometer
μm	micrometer
cm ⁻¹	Wavenumber (reciprocal centimeter)
λ	Wavelength (lambda)
Å	Molecular size (angstroms)
M	Molarity
mL	milliliter
m ³	Volume
cm ²	Surface area
$\mu\text{mHg/min}$	evacuation rate

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LIST OF ABBREVIATIONS

ATR	attenuated total reflectance
BET	brunauer–emmett–teller
CA	carbon aerogel
CMC	carboxymethyl cellulose
COVID	coronavirus disease
COF	carbon organic framework
CNT	carbon nanotube
DP	degree of polymerisation
FESEM	field emission scanning electron microscopy
FTIR	fourier transform infrared
FWHM	full width half maxima
FCV	fuel cell vehicle
IEA	international energy agency
MOF	metal organic frameworks
TPD	temperature programmed desorption
Mtep	million de tonnes équivalent pétrole (million tonne of oil equivalent)
QMS	quadrupole mass spectrometer
RSD	relative standard deviation
R/F	resorcinol/formaldehyde
SSA	specific surface area
TMP	turbomolecular pumps
XRD	X-ray diffraction
3D	three-dimensional