SUPERCAPATTERY PERFORMANCE OF MXENE-BASED COMPOSITE CONTAINING HKUST-1 AND HKUST-1 DERIVED COPPER OXIDE

By

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Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the degree of Master of Science

December 2023

FS 2023 27

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

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Sustainable energy storage is very important to fulfil the energy demand and reduce the carbon footprint caused by the combustion of fossil fuels. The selection of materials that have good conductivity, high surface area and electrochemically active is very important to achieve high specific energy and specific power energy storage. Herein, a supercapattery device was fabricated using two promising nanomaterials, MXene and copper-based metal-organic framework known as HKUST-1 (HKUST: Hong Kong University of Science and Technology). Two-dimensional titanium carbide, $Ti_3C_2T_x$ is a member of MXene that benefits from its conductive layer and electroactive sites with functionalised surface. HKUST-1 is known for its high surface area and highly porous structure that is efficient for electrolyte ion diffusion. The composite of MXene/HKUST-1 was synthesised through ultrasonication. The characterisations of samples were studied through XRD, Raman, BET, and FESEM for their crystallinity, functional groups, surface area, and morphology, respectively. Meanwhile, the specific capacity (C_s) of hybridised MXene/HKUST-1 was evaluated through cyclic voltammetry and galvanostatic charge discharge. The obtained C_s of MXene/HKUST-1 electrode in 1 M LiOH was 201.6 C g^{-1} at 4 A g^{-1}. MXene/HKUST-1 (positive electrode) and activated carbon (AC-negative electrode) were assembled into a supercapattery device (MXene/HKUST-1//AC). The device achieved C_s, specific energy (*E*), and specific power (*P*) of 53.6 C g^{-1} at 0.5 A g^{-1}, 11.2 Wh kg^{-1}, and 750 W kg^{-1}, respectively with capacity retention of 94% over 5000 cycles. Subsequently, the electrochemical study was performed on the MXene and HKUST-1-derived copper oxide (MXene/H-CuO), obtained from the calcination of HKUST-1 at 550°C for 2 hours in the air. The C_s of MXene/H-CuO attained 314 C g^{-1} at 4 A g^{-1}, whereas the MXene/H-CuO//AC supercapattery device obtained 47.2 C g^{-1} at 0.4 A g^{-1}. The *E* and *P* for the device were 9.8 Wh kg^{-1} and 600 W kg^{-1}, respectively. After 5000 cycles, the device retained 74% of its capacity. The remarkable specific capacities and specific energies of the supercapattery devices were contributed by the high surface area of HKUST-1 and the high conductivity of MXene and H-CuO.

Keywords: HKUST-1, HKUST-1-derived CuO, MXene, supercapattery
SDG: GOAL 7: Affordable and Clean Energy

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

PRESTASI SUPERKAPATERI BERASASKAN KOMPOSIT MXENE MENGANDUNGI HKUST-1 DAN KUPRUM OKSIDA TERBITAN HKUST-1

Oleh

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Penyimpanan tenaga mampan adalah sangat penting untuk memenuhi permintaan tenaga dan mengurangkan jejak karbon yang disebabkan oleh pembakaran bahan api fosil. Pemilihan bahan yang mempunyai kekonduksian yang baik, luas permukaan yang tinggi dan aktif secara elektrokimia adalah sangat penting untuk mencapai tenaga spesifik dan kuasa spesifik yang tinggi. Oleh itu, peranti superkapateri telah direka menggunakan dua bahan nano iaitu MXene dan rangka logam-organik (MOF) yang dikenali sebagai HKUST-1 (HKUST: Universiti Sains dan Teknologi Hong Kong). Titanium karbida dua dimensi, $Ti_3C_2T_x$ merupakan sejenis MXene yang menghasilkan manfaat daripada lapisan konduktif dengan tapak elektroaktifnya dilengkapi kumpulan berfungsi. HKUST-1 terkenal dengan luas permukaan dan struktur berliang tinggi yang berkesan untuk peresapan ion elektrolit. Komposit MXene/HKUST-1 disintesis melalui teknik ultrasonik. Pencirian sampel telah dikaji melalui XRD, Raman, BET, dan FESEM untuk mengkaji kehabluran, kumpulan berfungsi, luas permukaan dan morfologi. Manakala, kapasiti spesifik (C_s) MXene/HKUST-1 terhibrid dinilai melalui voltammetri kitaran dan galvanostatik cas-nyahcas. C_s elektrod MXene/HKUST-1 yang diperolehi dalam 1 M LiOH ialah 201.6 C g^{-1} pada 4 A g^{-1}. MXene/HKUST-1 (elektrod positif) dan karbon teraktif (AC-elektrod negatif) telah dipasang sebagai peranti superkapateri (MXene/HKUST-1//AC). Peranti mencapai C_s, tenaga spesifik (*E*) dan kuasa spesifik (*P*) masing-masing sebanyak 53.6 C g^{-1} pada 0.5 A g^{-1}, 11.2 Wh kg^{-1}, dan 750 W kg^{-1}. Manakala kapasiti yang dapat dikekalkan adalah sebanyak 94% selepas 5000 kitaran cas-nyahcas. Seterusnya, kajian elektrokimia telah dilakukan kepada komposit MXene dan kuprum oksida terbitan HKUST-1 (MXene/H-CuO). Kuprum oksida (H-CuO) diperoleh daripada pengkalsinan HKUST-1 pada 550°C selama 2 jam dalam udara. C_s MXene/H-CuO mencapai 314 C g^{-1} pada 4 A g^{-1}, manakala peranti superkapateri MXene/H-CuO//AC memperoleh 47.2 C g^{-1} pada 0.4 A g^{-1}. *E* dan *P* untuk peranti tersebut ialah masing-masing 9.8 Wh kg^{-1} dan 600 W kg^{-1}. Selepas 5000 kitaran, peranti mengekalkan 74% kapasiti asal. Nilai C_s dan *E* yang telah ditambah baik dipengaruhi oleh luas permukaan HKUST-1 yang tinggi dan kekonduksian tinggi MXene dan H-CuO.

Kata kunci: CuO terbitan HKUST-1, HKUST-1, MXene, superkapateri
SDG: MATLAMAT 7: Tenaga yang Berpatutan serta Bersih

ACKNOWLEDGEMENT

All praise and thanks be to Allah for the knowledge, understanding, strength, mercy and guidance along the journey.

My utmost gratitude and thanks to my supervisor, Assoc. Prof. Dr. Yusran Sulaiman for his supervision, guidance and support in every way. I would like to thank my co-supervisor, Assoc. Prof. Dr. Jaafar Abdullah for his helpful comments and suggestions. Millions of thanks to my senior lab mates: Dr. Hawa Nabilah Azman, Dr. Norhaffis Mustafa, Dr. Amirul Aizat Abdah, Dr. Dharshini Mohanadas, Logeswary Fiter and Velan Raman for their countless help, guidance, and support from day one of my research journey. A special thanks to the Scholarship Division, Ministry of Education Malaysia for the award of a fully paid study suit with a scholarship, administrators, lecturers, and staff of Institut Pendidikan Guru Kampus Pendidikan Teknik for their support and help with my service during the study period.

Finally, a special appreciation to my husband, Abd Muhaimin Amiruddin for his endless support and guidance, my mother Mrs. Arbaenah Maamun, my parents-in-law: Mr. Amiruddin Fakih and Mrs. Fatimah Kamaluddin, my children: Muna Safiyyah Abd Muhaimin, Maryam Abd Muhaimin, and Muhammad Muhsin Abd Muhaimin and all of my family members and friends for their help, motivation and prayers for me.

This thesis was submitted to the Senate of University Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AC	Activated carbon
BET	Brunauer-emmett-teller
BTC	1,3,5-benzenetricarboxylate
BTC	Benzene-1,3,5-tricarboxylate
CFP	Carbon fibre paper
CNT	Carbon nanotubes
CNT	Carbon nanotubes
CNTF	Carbon nanotube film
CPE	Carbon paste electrode
CTAB	Cetyltrimethyl ammonium bromide
CV	Cyclic voltammetry
DI	Deionised
DMF	Dimethylformamide
DMSO	Dimethyl sulfoxide
DSBDC	2,5-dimercaptoterephthalate
EDLC	Electric double-layer capacitors (EDLC),
EDX	Energy dispersive X-ray
EES	Electrochemical energy storage
EIS	Electrochemical impedance spectroscopy
EIS	Electrochemical impedance spectroscopy
FESEM	Field emission scanning electron microscopy
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared

GCD	Galvanostatic charge-discharge
H3BTC	Trimesic acid
H4DSBDC	2,5-dihydroxybenzene-1,4-dicarboxylic acid
H-CuO	HKUST-1-derived cuo
HITP	2,3,6,7,10,11-hexaiminotriphenylene
HKUST-1	Hong Kong University of Science and Technology-1
ITO	Indium tin oxide
LA	Lauric acid
LDH	Layered double hydroxide
LIB	Lithium-ion batteries
MOF	Metal-organic framework
NF	Nickel foam
NMR	Nuclear magnetic resonance
PANI	Polyaniline
PEDOT	Poly(3,4-ethylenedioxythiophene)
PPy	Polypyrrole
PrGO	Poly(3,4-ethylenedioxythiophene)-reduced graphene oxide
PSM	Post-synthetic modification
PTFE	Poly(tetrafluoroethylene)
PTFE	Polytetrafluoroethylene
PVA	Poly(vinyl alcohol)
PVDF	Polyvinylidene fluoride
rGO	Reduced graphene oxide
rHGO	Reduced holey graphene network
SBU	Secondary building unit

TCNQ	Tetracyanoquinodimethane
TMS	Transition metal sulphide
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Research Background

In the midst of Industrial Revolution 4.0, technology has become part and parcel of our daily life. Recently, robust energy storage has become a demand for hybrid vehicles, electric transportation, and portable electronic devices. A compact, lightweight, fast charging, able to supply energy for a long time and durable energy storage (Anasori et al., 2017, Melchior et al., 2018) has given great value to the practicality of the devices. Energy storage is also very essential in storing the electrical energy generated from intermittent renewable energy sources such as solar, tidal, wave and wind (Balasubramaniam et al., 2020). Efficient energy storage is vital to enhance the use of renewable energy as nowadays fossil fuels have taken their toll on the environment causing large-scale issues such as terrible climate change.

Electrochemical energy storage (EES) uses chemical energy to generate electricity which is now a great solution to the energy and environmental crisis of this century. Among the classes of EES, supercapacitors and batteries gain massive attention to cater to the energy needs. Supercapacitors are better than batteries in the aspects of safety (Wang et al., 2018d), life cycle (Cossutta et al., 2020) and power (Mostazo-López et al., 2018) but battery produces higher specific energy (Libich et al., 2018). The combination of supercapacitor and battery, termed supercapattery, has gained attention for the development of safe, long cycle life and high-performance energy storage with high specific power and specific energy (Chen, 2017, Fujishige et al., 2017a). Supercapatteries can also achieve a range of energy and power comparable to or exceeding lithium-ion batteries (LIB) (Libich et al., 2018).

1.2 Problem Statement

Batteries and supercapacitors are the two types of energy storage systems. Compared to batteries, supercapacitors have longer cycle life, rapid charge-discharge rate, high specific power as well as cost-efficient (Dubal et al., 2018, Ramachandran et al., 2018b). However, the drawback that restricts the use for practical application is unsatisfactory specific energy which can be less than 10 Wh kg^{-1} (Ma et al., 2021). Meanwhile, batteries are known to have high specific energy but low specific power (Pal, 2019) and are less environmentally friendly (Khan et al., 2020). Therefore, the merits of supercapacitors with specific power and battery's specific energy could be achieved by combining the supercapacitor electrode and battery electrode in a device known as a supercapattery (Zuo et al., 2017).

Technically, the specific energy of a supercapacitor is influenced by specific capacitance and potential windows (Balasubramaniam et al., 2020). One of the promising materials for delivering high capacitance is MXene, such as titanium carbide ($Ti_3C_2T_x$). As a 2D material, MXene contains layers with large surface area, exposed surface atoms, and Van der Waals forces at the layer edge to facilitate the intercalation of electrolyte ions in between the layers (Nasrin et al., 2021) and metallic conductivity (Shahzad et al., 2016). The electrochemical performance can be boosted through fast electron transfer due to good electrical conductivity. There is also a smaller volume change during ion intercalation and de-intercalation caused by the large interlayer spacings (Ali et al., 2021). Nonetheless, the main challenge that comes along with the benefits is the poor structural stability of MXene. Pristine MXene suffers from self-restacking and is easily oxidised, impacting the reduction of the active sites, restraining the redox reaction on the MXene surface, lowering the conductivity, and therefore impeding the electrochemical performance (Zang et al., 2020).

HKUST-1 (Hong Kong University of Science and Technology-1) or MOF-199 is one of the most explored metal-organic frameworks (MOFs) for application in the electrochemical energy storage (EES) field. Like the other members of MOF, HKUST-1 is known for its high porosity, versatile functionalities, variety of structures and controllable chemical compositions that are favourable in increasing the electrochemical performance of EES devices (Zhao et al., 2018). Despite the desirable criteria, MOFs' application is challenged by the low structural stability and electrical conductivity (Xie et al., 2020). Therefore, numerous research works are dedicated to fully utilising the benefits of HKUST-1.

To attain the structural stability of MXene and the electrical conductivity of MOFs, it is brilliant to integrate the two materials as a composite to be used in the supercapattery device. The hybridization with MOF is expected to prevent the self-restacking of MXene layers (Nasrin et al., 2021). The formation of interlayer hydrogen bonding (Liu et al., 2020c) or electrostatic interaction (Wang et al., 2020a) between the MXene sheets and MOFs could hinder the Van der Waals attraction among the MXene layers that cause the self-restacking (Ran et al., 2020). Meanwhile, the interconnected porous structure of MOF could enhance the ion transfer through the electrode material (Xie et al., 2020). The synergistic effect between MXene and MOF could improve the charge storage and rapid charge transfer process, hence enhancing the electrochemical performance of the supercapattery.

Metal oxides are well-known materials for pseudocapacitor application (Du et al., 2018). Copper oxide (CuO) has gained a lot of attention due to its environmental friendliness (Wu et al., 2013), abundance, non-toxic, low cost (Majumdar and Ghosh, 2021), high thermal and chemical stability, easy synthesis, and high theoretical capacitance (Zhu et al., 2022). Recently, metal oxides derived from MOFs have been explored as a way to surmount MOFs' poor conductivity which is synthesised through a simple heat treatment (Banerjee et al., 2013). The porosity and large surface area of metal oxides facilitate fast ion and electron transport through effective contact between the electrolyte and electrode materials (Wang et al., 2015a). However, the electrochemical properties of HKUST-1-derived CuO remain inadequate (He et al., 2018) for many applications including supercapattery due to relatively low electrical conductivity and poor stability (Zhi et al., 2015). Therefore, in the second part of this research, the electrically conductive MXene will be hybridized with HKUST-1-derived CuO to tune the active sites of CuO and promote the electron transport pathways (Murphin Kumar et al., 2020). The synergistic effect could enhance the electrochemical performance and the stability of CuO while providing support to prevent MXene agglomeration.

1.3 Objectives of the study

The aim of this research is to fabricate MXene/HKUST-1 and MXene/HKUST-1-derived CuO (MXene/H-CuO) for the supercapattery application with improved electrochemical performance compared to the pristine MXene, HKUST-1 and H-CuO. In an effort to achieve this purpose, the research outlines three specific objectives:

1. To prepare MXene/HKUST-1 and MXene/H-CuO electrodes.
2. To study the physicochemical characteristics of MXene/HKUST-1 and MXene/H-CuO composites.
3. To fabricate and evaluate the supercapattery performance of MXene/HKUST-1 and MXene/H-CuO electrodes.

1.4 Scope and limitation of the study

This research focuses on the electrochemical performance of the electrode materials of MXene, HKUST-1, HKUST-1-derived CuO (H-CuO), the composites of MXene/HKUST-1 and MXene/H-CuO in 1 M LiOH electrolyte. The as-synthesised composites are studied for their morphology and distribution of the elements, phase and crystallinity, vibrational modes, surface area and porosity as well surface chemical composition of the composites that are responsible for the electrochemical performance. The physicochemical characterisations were performed using field emission scanning electron microscope (FESEM), energy dispersive X-ray (EDX), X-ray diffraction (XRD), Raman spectroscopy, Brunauer-Emmett-Teller *(*BET*)* method and X-ray photoelectron spectroscopy (XPS).

The electrochemical properties of MXene/HKUST-1 and MXene/CuO were evaluated based on the specific capacity of the composites as a working electrode against Ag/AgCl reference electrode within an optimized potential window, scan rates and current densities throughout the cyclic volumetric (CV) and galvanostatic charge-discharge (GCD) test. The supercapattery performance of the composite materials was subsequently evaluated as the positive electrode in a supercapattery device (two-electrode system) with the activated carbon as the negative electrode. The assessments were performed through CV, GCD, electrochemical impedance spectroscopy (EIS) and stability test. Consequently, the specific capacity, resistance, capacity retention, specific energy and specific power were determined.

Limitations of the study are described as follows:

1. The properties of materials are very much influenced by the synthesis method. In this study, the characterisations of MXene will only focus on $Ti_3C_2T_x$ which was synthesised through chemical etching using 9 M HCl and LiF at 40°C (48 h). This choice was made considering the safety issues of conventional hydrogen fluoride, HF etchant and the successive $Ti_3C_2T_x$ obtained from this etching process. This method of synthesis also has to deal with the incomplete removal of Al from Ti_3AlC_2 which might affect the electrochemical properties of MXene produced.
2. The simultaneous oxidation behaviour possessed by MXene is also a limitation of this study. In this case, $Ti_3C_2T_x$ is prone to be oxidized into TiO_2. As much care should have been taken there is still a chance of oxidation can happen such as exposure to the air and humidity during transferring the MXene solids, reaction with dissolved oxygen in deionized water during the washing process and while storing the MXene. This will affect the purity of MXene over time and slightly inconsistent data might not be obtained with the same batch of MXene. Due to this, the MXene was synthesised in a small portion. Next, the vial containing dried MXene powder was sealed with a parafilm and purged with nitrogen gas to eliminate oxygen before screwing the cap tightly for storage. It is important to be cautious of the storage period as complete

oxidation might happen within four weeks without any visible changes (Habib et al., 2019).

1.5 Outline of the thesis

Chapter 1 discusses the research background, problem statements, objectives, scope and limitations of the study. Chapter 2 provides reported works related to the scope of the study which are supercapattery, MXene, HKUST-1 and HKUST-1-derived copper oxides. Chapter 3 presents the methodology of materials preparation, and physicochemical characterisation, followed by the fabrication of the electrodes for electrochemical characterisation and finally, the assembly techniques of the supercapattery devices to evaluate the electrochemical performance. Chapter 4 explains the results of physicochemical and electrochemical characterisation. Chapter 5 concludes the study findings and recommendations for future improvement to this work.

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