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DEVELOPMENT OF HIGH-PERFORMANCE ELECTROCHROMIC SUPERCAPACITORS USING COPPER-BASED METAL-ORGANIC FRAMEWORK/REDUCED GRAPHENE OXIDE COMPOSITES

DHARSHINI A/P MOHANADAS

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By

DHARSHINI A/P MOHANADAS

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Chair: Associate Professor Yusran Sulaiman, PhD
Faculty: Science

Supercapacitors (SCs) are promising energy storage devices with tremendous specific capacitance, fast rate for charging/discharging, high specific power and long-life cycle. It is difficult to estimate the state-of-charge of the existing supercapacitors, which leads to excessive charging/discharging. Hence, supercapacitors with more functions and novel features are fabricated to widen the energy storage applications. The electrochromic (EC) materials are able to exhibit visual color changes upon an applied potential. Therefore, it is highly attractive to combine SC and EC characteristics into one device for bifunctional applications namely electrochromic supercapacitor (EC-SC). The EC-SCs can function as a normal supercapacitor to store energy, at the same time, the energy level of the devices can be sensed by observing the changes in visual colors. In this work, copper-based metal-organic framework/reduced graphene oxide (HKUST-1/rGO) composites were successfully developed as high-performance EC-SC devices via hydrothermal and electrochemical deposition. The as-fabricated composites were characterized via X-ray diffraction (XRD), Fourier transform infrared spectrometry (FTIR), Raman spectroscopy, field emission scanning microscopy (FESEM) and electrochemical impedance spectroscopy (EIS). Initially, poly(3,4-ethylenedioxythiophene)-reduced graphene oxide/copper-based metal-organic framework (PEDOT-rGO/HKUST-1) was prepared and electrochromic performance was evaluated. PEDOT-rGO/HKUST-1 exhibited a high coloration efficiency of 176.8 cm^2/C with high optical contrast. An electrochromic coloration and bleaching times of 1.1 s and 1.3 s were recorded at 500 nm which proved excellent switching kinetics of PEDOT-rGO/HKUST-1. The similar composite also depicted promising supercapacitive properties with remarkable specific energy and specific capacitance of 21.0 Wh/kg and 360.5 F/g, respectively. The HKUST-1/rGO composite was designed as a negative electrode of an asymmetrical supercapacitor (ASC) device. A vanadium oxide/reduced graphene oxide//copper-based metal-organic framework/reduced graphene oxide (V_2O_5/rGO//HKUST-1/rGO) ASC device successfully delivered enormous specific energy (31.2 Wh/kg) at a specific power of 656.4 W/kg. Nickel oxide/vanadium oxide/reduced graphene oxide//HKUST-1/rGO (NiO/V_2O_5/rGO//HKUST-1/rGO) and manganese oxide/ vanadium oxide/reduced graphene oxide//HKUST-1/rGO (MnO_2/V_2O_5/rGO//HKUST-1/rGO) devices were developed for bifunctional asymmetrical electrochromic supercapacitor (EC-SC) devices. The designed PEDOT-rGO/HKUST-1, V_2O_5/rGO//HKUST-1/rGO, NiO/V_2O_5/rGO//HKUST-1/rGO and MnO_2/V_2O_5/rGO//HKUST-1/rGO depicted outstanding specific capacitance of 360.5, 483.9, 500 and 652.7 F/g, respectively. Both bifunctional asymmetric EC-SC devices (NiO/V_2O_5/rGO//HKUST-1/rGO and MnO_2/V_2O_5/rGO//HKUST-1/rGO) were also demonstrated green and orange color at the discharged and charged state, respectively. These results proved that the successfully prepared asymmetrical EC-SC devices with both excellent charge storage capability and outstanding electrochromic properties are promising candidates for bifunctional application.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN ELEKTROKROMIK SUPERKAPASITOR BERPRESTASI TINGGI MENGGUNAKAN KOMPOSIT KERANGKA LOGAM-ORGANIK BERASASKAN KUPRUM/GRAFIN OKSIDA TERTURUN

Oleh

DHARSHINI A/P MOHANADAS

September 2021

Pengurusi: Prof Madya Yusran Sulaiman, PhD
Fakulti: Sains

Superkapasitor (SC) adalah alat penyimpanan tenaga yang mempunyai kapasitan spesifik yang tinggi, kadar caj/discaj yang pantas, kuasa spesifik yang tinggi dan kitaran jangka hayat yang panjang. Ia adalah sukar untuk menentukan keadaan caj di dalam superkapasitor yang sedia ada yang menyebabkan caj/discaj yang berlebihan. Oleh itu, superkapasitor dengan lebih banyak fungsi dan ciri baru direka untuk memperluaskan aplikasi penyimpanan tenaga. Bahan elektrokromik (EC) boleh menunjukkan perubahan warna visual pada potensi yang diaplikasikan. Ia sangat menarik untuk mengintegrasikan ciri-ciri SC dan EC ke dalam satu alat dwifungsi yang dikenali sebagai elektrokromik superkapasitor (EC-SC). EC-SCs berfungsi sebagai superkapasitor yang biasa untuk menyimpan tenaga, pada masa yang sama, tahap tenaga alat dapat dikesan dengan memerhatikan perubahan warna visual. Dalam kerja ini, kerangka logam-organik berasaskan kuprum/grafin oksida terturun (HKUST-1/rGO) komposit telah berjaya dihasilkan sebagai EC-SC yang berprestasi tinggi melalui hidroterma dan pengendapan elektrokimia. Komposit-komposit yang dihasilkan telah dicirikan menggunakan belauan sinar-X (XRD), spektroskopi Fourier inframerah (FTIR), spektroskopi Raman, medan pancaran mikroskopi imbasan elektron (FESEM), spektroskopi fotoelektron sinar-X (XPS) dan spektroskopi elektrokimia impedan (EIS). Pada awalnya, poli(3,4-etilenadioksitiofena)-grafin oksida terturun/kerangka logam-organik berasaskan kuprum (PEDOT-rGO/HKUST-1) telah dihasilkan dan prestasi elektrokromik telah dinilai. PEDOT-rGO/HKUST-1 telah menunjukkan kecekapan pewarnaan 176.8 cm^2/C dengan kontras optik yang tinggi. Masa pewarnaan 1.1 s dan pelunturan 1.3 s elektrokromik telah direkodkan pada 500 nm membuktikan penukaran kinetik PEDOT-rGO/HKUST-1 yang cemerlang. Komposit yang sama

juga menunjukkan sifat superkapasiti yang menjanjikan dengan tenaga spesifik 21.0 Wh/kg dan kapasitan spesifik 360.5 F/g yang luar biasa. HKUST-1/rGO komposit direka sebagai elektrod negatif alat superkapasitor asimetri (ASC). Alat ASC vanadium oksida/grafin oksida terturun//kerangka logam-organik berasaskan kuprum/grafin oksida terturun (V_2O_5/rGO//HKUST-1/rGO) berjaya menyampaikan tenaga spesifik (31.2 Wh/kg) yang tinggi pada kuasa spesifik 656.4 W/kg. Nikel oksida/vanadium oksida/grafin oksida terturun//kerangka logam-organik berasaskan kuprum/grafin oksida terturun (NiO/V_2O_5/rGO//HKUST-1/rGO) and mangan oksida/vanadium oksida/grafin oksida terturun//kerangka logam-organik berasaskan kuprum/grafin oksida terturun (MnO_2/V_2O_5/rGO//HKUST-1/rGO) dijadikan alat dwifungsi elektrokromik-superkapasitor (EC-SC) asimetri. PEDOT-rGO/HKUST-1, V_2O_5/rGO//HKUST-1/rGO, NiO/V_2O_5/rGO//HKUST-1/rGO dan MnO_2/V_2O_5/rGO//HKUST-1/rGO yang direka menunjukkan kapasitan spesifik masing-masing yang tinggi iaitu 360.5, 483.9, 500 and 652.7 F/g. Alat-alat EC-SC asimetri (NiO/V_2O_5/rGO//HKUST-1/rGO dan MnO_2/V_2O_5/rGO//HKUST-1/rGO) juga menujukkan warna hijau dan oren masing-masing pada keadaan dinyahcaj dan dicaj. Hasil kajian ini membuktikan bahawa alat-alat EC-SC asimetri yang berjaya disediakan dengan keupayaan penyimpanan caj yang luar biasa dan sifat elektrokromik yang cemerlang merupakan calon yang sangat sesuai untuk aplikasi dwi-fungsi.

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Yusran Bin Sulaiman, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Jaafar Bin Abdullah, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

Nor Azam Bin Endot, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

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Professor and Dean
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Δt	Discharging time
ΔT	Optical contrast
ΔV	Potential window
AC	Activated carbon
ACN	Acetonitrile
ASC	Asymmetrical supercapacitor
BDC	Terephthalic acid
BMOs	Bimetallic oxides
BTC	Trimesic acid
CA	Chronoamperometry
CE	Coloration efficiency
CFP	Carbon fiber paper
CNF	Carbon nanofiber
CPs	Conducting polymers
C_{sp}	Specific capacitance
CV	Cyclic voltammetry
E	Specific energy
EC	Electrochromic
EC-SC	Electrochromic supercapacitor
EDLC	Electrochemical double layer capacitor
EDOT	3,4-Ethylenedioxythiophene
E_g	Energy band gap
EIS	Electrochemical impedance spectroscopy

ESR	Equivalent series resistance
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
FTO	Fluorine-doped tin oxide
GCD	Galvanostatic charge discharge
GO	Graphene oxide
GQDs	Graphene quantum dots
HKUST-1	Copper-based MOF
ITO	Indium tin oxide
LBL	Layer-by-layer
LMCT	Ligand to metal charge transfer
MnO_x	Manganese oxide
MOF	Metal-organic framework
MWCNTs	Multi-walled carbon nanotubes
$NiCo_2O_4$	Nickel cobalt oxide
Ni-MOF	Nickel-based MOF
NiO_x	Nickel oxide
NMP	N-methylpyrrolidone
OCP	Open circuit potential
OD	Optical density
P	Specific power
P5Fin	Poly(5-formylindole)
P6ICA	Poly(indole-6-carboxylicacid)
PANI	Polyaniline

PEDOT	Poly(3,4-ethylenedioxythiophene)
PEDOT:PSS	Poly(styrenesulfonate):poly(3,4-ethylenedioxythiophene)
PET	Polyethylene terephthalate
PI	Polyimide
PPy	Polypyrrole
Pt	Platinum
PVDF	Polyvinylidene fluoride
Q_d	Charge density
R_{ct}	Charge transfer resistance
RE	Reference electrode
rGO	Reduced graphene oxide
SC	Supercapacitor
SWCNTs	Single-walled carbon nanotubes
t_b	Bleaching time
TBAP	Tetrabutylammonium perchlorate
t_c	Coloration time
TMOs	Transition metal oxides
UV	Ultraviolet-visible
v	Scan rate
VO_x	Vanadium oxide
WE	Working electrode
WO_x	Tungsten oxide
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

λ Wavelength

CHAPTER 1

INTRODUCTION

1.1 Background of research

Over the past few decades, the world population and the global energy demand are increasing rapidly. The main source of energy is obtained from non-renewable fossil fuels such as coal, oil, and natural gas. The high consumption of fossil fuels causes rapid depletion of resources. Moreover, the high usage of non-renewable sources of energy results in environmental pollutions i.e., global warming, climate change and ozone layer depletion. On a global scale, the demand for renewable energy is extremely high in order to lower the dependency on non-renewable energy resources and to meet the high-power demand. Solar energy, wind energy, geothermal energy, bioenergy, hydro energy and marine energy are the well-known primary renewable sources (Mahian *et al.*, 2021). According to the U.S Energy Information Administration (EIA), 23% of the total electricity of the world was supplied from renewable energy sources in 2015. The demand for energy from renewable resources gradually elevated from 26% (2019) to 28% (2020) due to the Covid-19 pandemic and it is expected to increase up to 30% in 2022 (IEA, 2020).

Various electrical energy storage systems such as supercapacitors, batteries and fuel cells have been developed extensively to fulfill the needs of customers. Supercapacitors are environment-friendly energy storage devices with high-rate capability, great specific power, and good cyclability (Ma *et al.*, 2021). The supercapacitive performance of an energy storage device is highly dependent on the selection of the electroactive material. Three criteria should be taken into consideration for a high-performance supercapacitor electrode, which are a vast surface area for efficient charge storage, promising electroconductivity for good ion transmission and superior cyclability for environmental protection (Xiong *et al.*, 2020).

Since the usage of portable and wearable electronic devices is extremely high nowadays, multi-functions and smart features are introduced in energy storage devices. The bifunctional electrochromic supercapacitors (EC-SCs) are being explored because it has the ability to store energy as a traditional supercapacitor, at the same time, the energy level in the device can be easily identified based on visual color changes. Moreover, this additional functionality in an energy storage device can effectively overcome the damage caused by excessive charging/discharging. Therefore, developing high-performance EC-SCs are highly needed to widen the energy storage application.

1.2 Problem statement

Metal-organic frameworks (MOFs) are the porous crystalline materials that are widely explored and labeled as the most promising materials in supercapacitors due to their high internal pore volume, high surface area and good thermal stability. However, MOFs have poor stability and electroconductivity properties, which limits the energy storage performance. This issue can be overcome by incorporating MOFs with carbon-based materials (e.g., graphene oxide (GO), reduced graphene oxide (rGO), multi-walled carbon nanotubes (MWCNTs), carbon nanofibers (CNFs)) that have high mechanical strength and electrical conductivity.

Besides supercapacitive performance, it is difficult to estimate the state-of-charge of the existing supercapacitors, which leads to excessive charge/discharge. Hence, supercapacitors with more functions and novel features are being pursued to overcome this issue and also to widen the energy storage applications. Electrochromism is a phenomenon related to reversible color changes, which caused by the electrochemical redox reaction. Generally, the electrochromic materials consume power during the color switching, and the required switching voltage is usually very small. The electrochromic color variation is mainly determined by the ion intercalation and deintercalation processes. This electrochemical mechanism is similar to the operating principle of an electrochemical supercapacitor. Therefore, it is highly attractive to combine supercapacitive and electrochromic characteristics into one device for bifunctional applications namely EC-SC. The EC-SC can potentially operate as a normal SC to store electrical energy, at the same time, the energy level can be determined by demonstrating changes in the visual colors upon the applied potential.

For easy estimation of energy storing states, the color of active materials used should be easily sensed by the human. There are few potential transition metal oxides (TMOs i.e., V_2O_5. NiO, MnO_2) and conducting polymers (CPs i.e., PEDOT, PANI, PPy) that can serve as active materials for both supercapacitors and electrochromic devices because they are capable to store energy and also can depict obvious color changes, utilizing fast-faradic reactions. However, the poor electrical conductivity of TMOs and poor cyclic stability of CPs could significantly impact the rate capability and cycling stability of the material. This issue can be overcome by producing low-cost bimetallic oxides (BMOs), which consist of mixed metal elements. BMOs with multiple oxidation states and superior electroactive sites could elevate the capacitive property and cycling stability of the device. The BMOs theoretically have good optical characteristics that could successfully demonstrate visual color changes upon the charging/discharging process. Therefore, in this study, copper-based MOF (HKUST-1), rGO and BMO-based materials are used for the fabrication of asymmetrical EC-SC devices.

1.3 Objectives

The purpose of this research is to design, fabricate and investigate the HKUST-1/rGO-based composites for bifunctional EC-SC applications. Following are the detailed objectives of this research:

i. To prepare and study electrochromic performance of poly(3,4-ethylenedioxythiophene)-reduced graphene oxide/copper-based metal-organic framework (PEDOT-rGO/HKUST-1).

ii. To evaluate supercapacitive performance of PEDOT-rGO/HKUST-1.

iii. To fabricate high-performance HKUST-1/rGO composites as a negative electrode of an asymmetrical supercapacitor device.

iv. To evaluate both electrochromic and supercapacitive performance of multicolored NiO/V_2O_5/rGO//HKUST-1/rGO and MnO_2/V_2O_5/rGO//HKUST-1/rGO EC-SC devices.

1.4 Scope of study

This study emphasizes the fabrication of HKUST-1/rGO-based composites as a high-performance bifunctional EC-SC with promising energy storing (enormous specific capacitance, great specific energy and high specific power and excellent cyclability) and good optical (prominent color changes, high coloration efficiency, rapid switching kinetics) properties. Firstly, a PEDOT-rGO/HKUST-1 was electrochemically prepared and its electrochromic performance was evaluated. The hydrothermally prepared HKUST-1 was then integrated with the PEDOT-rGO and it was developed as a supercapacitor device to identify the energy storage capability of the PEDOT-rGO/HKUST-1. The operating potential of the device was widened by introducing an ASC, highlighting the HKUST-1/rGO as the negative electrode of the ASC device. Next, the electrochromic and supercapacitive characteristics were integrated by combining optically active bimetallic oxides (NiO/V_2O_5 and MnO_2/V_2O_5) with the rGO, which were used as the positive electrodes of the asymmetrical EC-SC (NiO/V_2O_5/rGO//HKUST-1/rGO and MnO_2/V_2O_5/rGO//HKUST-1/rGO) devices. The conceived high-performance asymmetrical EC-SC devices were examined via electrochemical and electrochromic measurements.

1.5 Hypothesis

Considering the problems in this research, few hypotheses have been made:

i. The proper selection of electroactive material is able to enhance the energy storage properties as well as electrochromic characteristics. The selected electrodes such as HKUST-1, rGO and BMO possess vast surface area, providing great electrolyte ion accessibility for enhanced faradaic redox reaction.

ii. The promising electrical conductivity of the electrode mainly reduces the internal resistance of the device. The fabrication of BMO that consists of mixed metal elements and multiple oxidation states promotes the electroactive sites for an excellent electron transfer process.

iii. An electroactive material with great mechanical strength exhibits a long-life cycle for environmental protection. The inclusion of rGO with great mechanical strength increases the electrochemical stability of EC, SC and EC-SC materials.

iv. The anodic coloration materials and cathodic coloration materials are the suitable positive and negative electrodes of EC or EC-SC, respectively. The electrochemically conductive PEDOT capable to display color changes at the negative potential. NiO, V_2O_5 and MnO_2 are anodic coloration TMOs that can be fabricated as the positive electrode of the bifunctional device to achieve both color changing and energy-storing properties.

1.6 Organization of chapters

This thesis comprises eight chapters and these chapters are arranged as follows. The introduction of this study is described in Chapter 1, which consists of the background of the research, problem statement, objectives, and scope of the study. The recent developments of CP/TMO-, graphene- and MOF-based composites for electrochromic, supercapacitor and bifunctional EC-SC applications are discussed in Chapter 2. The materials and instrumentations that utilized for the HKUST-1/rGO-based composites are elaborated in Chapter 3. The electrochromic performance of PEDOT-rGO/HKUST-1 is explained in Chapter 4. Chapter 5 elaborates on the supercapacitive performance of the PEDOT-rGO/HKUST-1. The electrochemical performance of HKUST-1/rGO at the negative potential is discussed in Chapter 6. Chapter 7 demonstrates NiO/V_2O_5/rGO (positive electrode) for a bifunctional asymmetrical EC-SC application. The electrochemical and electrochromic characteristics of the bifunctional MnO_2/V_2O_5/rGO//HKUST-1/rGO device are discussed in Chapter 8. The summary, conclusion and recommendations for future works are described in Chapter 9.

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BIODATA OF STUDENT

Dharshini A/P Mohanadas was born in Hospital Banting on 15th February 1995. She had her earlier primary education at Sekolah Jenis Kebangsaan (Tamil) Jugra and completed her secondary school studies at Sekolah Menengah Kebangsaan Bandar Banting. In 2013, she was offered to continue her studies at Kolej Matrikulasi Pahang. Then, she learned (2014-2018) degree in Bachelor of Science (Honours) Major Chemistry at Universiti Putra Malaysia (UPM). After completing the 1st degree studies, she chose to join a PhD program at UPM under the supervision of Assoc. Prof. Dr. Yusran Sulaiman.

LIST OF PUBLICATIONS

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