# ELECTROCHEMICAL PERFORMANCE OF AQUEOUS HYBRID SUPERCAPACITOR BASED ON LITHIUM IRON PHOSPHATE/SILICON/GRAPHENE COMPOSITE

**By**

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**Chair : Professor Janet Lim Hong Ngee, PhD**
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Aqueous hybrid supercapacitors (HS) are a viable alternative to achieve low-cost, environmentally friendly, and safer energy storage technologies. Herein, lithium iron phosphate modified silicon and graphene derivatives (LFP/Si/graphene) are constructed as the cathode and graphene nanoplatelets (GNPs) as the anode for an aqueous HS. The incorporation of GNPs and reduced graphene oxide (rGO), which is the source of graphene derivatives (graphene) in the composite, has played the role of an electronic conductivity enhancer for LFP and a binder for Si in the framework structure of the rGO film. Therefore, Si is able to perform its role effectively in avoiding surface instability issues and preventing the side reaction of LFP in an aqueous electrolyte. The electrochemical performance is evaluated in a 1 M $Li_2SO_4$ aqueous electrolyte that has been thoroughly deaerated with high-purified argon (Ar) gas to remove any dissolved oxygen that might interfere with the analysis. The Ar blanketing in the electrolyte intensified the electrochemical performance by 60% as compared to the Ar-free electrolyte, proving that the presence of Ar has offered a more stable electrolyte to protect the electrode surfaces from contaminants and impurities during charging and discharging processes. The coated LFP by 0.5 w/w% Si with incorporation of graphene ($LFP/Si_{0.5}$/graphene) for electronic conductivity enhancement in Swagelok-type cells has an exceptional specific capacitance of 272.6 F $g^{-1}$ and a highly improved cycle life, which shows 87.2% capacitance retention after 5200 cycles. Moreover, a different assembly of a close-to-current commercial format of roll-type cell is also studied, which exhibits a slightly higher specific capacitance of 301.3 F $g^{-1}$ with energy and power density of 35.62 Wh $kg^{-1}$ with 922.53 W $kg^{-1}$, respectively. Therefore, both cell configurations of Swagelok-type and roll-type, using $LFP/Si_{0.5}$/graphene, have displayed promising applications of the HS, which have the potential to slowly build themselves into a vital role in hybrid-electric technology.

**Keywords:** Aqueous hybrid supercapacitor; Ar blanketing; Lithium iron phosphate/silicon/graphene cathode; Roll-type cell; Swagelok-type cell

**SDG:** GOAL 4: Quality Education

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

**PRESTASI ELEKTROKIMIA SUPERKAPASITOR HIBRID AKUEUS YANG BERASASKAN KOMPOSIT LITIUM BESI FOSFAT/SILIKON/GRAFIN**

Oleh

**WAN NADHIRAH BINTI WAN MOHD ABD KALAM**

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Superkapasitor hibrid (HS) akueus adalah satu alternatif yang berdaya maju untuk mencapai teknologi penyimpanan tenaga yang berkos rendah, mesra alam, dan lebih selamat. Di sini, litium besi fosfat yang diubahsuai dengan silikon dan derivatif grafin (LFP/Si/graphene) dibina sebagai katod serta nanoplatelet grafin (GNPs) sebagai anod untuk HS akueus. Gabungan GNP dan grafin oksida terturun (rGO) yang menjadi sumber kepada derivatif grafin (graphene) telah memainkan peranan sebagai penambahbaik kekonduksian elektronik untuk LFP dan pengikat kepada Si dalam struktur rangka kerja filem rGO. Oleh itu, Si dapat melaksanakan peranannya dengan berkesan bagi mengelakkan masalah ketidakstabilan permukaan dan mencegah tindak balas sampingan pada LFP di dalam elektrolit akueus. Prestasi elektrokimia dinilai di dalam elektrolit akueus 1 M $Li_2SO_4$ yang dinyahudara secara teliti dengan gas argon (Ar) yang berketulenan tinggi untuk menghilangkan larutan oksigen yang akan mengganggu analisis. Selimutan Ar di dalam elektrolit telah meningkatkan prestasi elektrokimia sebanyak 60%

berbanding dengan elektrolit bebas Ar yang membuktikan bahawa kehadiran Ar telah memberikan elektrolit yang lebih stabil untuk melindungi permukaan-permukaan elektrod daripada bahan-bahan kontaminasi dan bendasing semasa proses pengecasan dan penyahcasan. LFP yang bersalut 0.5 w/w% Si dengan gabungan grafin (LFP/$Si_{0.5}$/graphene) yang memberi peningkatan kekonduksian elektronik pada sel jenis Swagelok mempunyai kapasitan spesifik yang luar biasa sebanyak 272.6 F $g^{-1}$ dan menunjukkan kitaran hidup yang sangat baik dengan 87.2% pengekalan kapasiti selepas 5200 kitaran. Selain itu, pemasangan yang berbeza juga dikaji dengan format jenis gulung yang seakan-akan dengan format komersial semasa, di mana ia mempamerkan kapasitan spesifik yang lebih tinggi iaitu 301.3 F $g^{-1}$ dengan ketumpatan tenaga dan kuasa masing-masing ialah 35.62 Wh $kg^{-1}$ dan 922.53 W $kg^{-1}$. Oleh itu, kedua-dua konfigurasi sel berjenis Swagelok dan gulung yang menggunakan LFP/$Si_{0.5}$/graphene telah memaparkan aplikasi HS yang berpotensi untuk dijadikan peranan penting dalam teknologi elektrik-hibrid.

**Kata Kunci:** Katod litium besi fosfat/silikon/grafin; Sel jenis gulung; Sel jenis Swagelok; Selimutan Ar; Superkapasitor hibrid akueus

**SDG:** Matlamat 4: Pendidikan Berkualiti

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This thesis was submitted to the Senate of Universiti 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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lower and (I) higher magnifications, and (J) EDX elemental analysis after 5200 cycles.

# LIST OF ABBREVIATIONS

| | |
|---|---|
| BET | Brunauer-Emmet-Teller |
| CFF | Carbon fabric cloth |
| CMPs | Conjugated microporous polymers |
| CNTs | Carbon nanotubes |
| CPE | Constant phase element |
| CV | Cyclic voltammetry |
| EDLC | Electrochemical double layer capacitors |
| EDX | Energy dispersive X-ray |
| EES | Electrical energy storage |
| EIS | Electrochemical impedance spectroscopy |
| FESEM | Field emission scanning electron microscope |
| GCD | Galvanostatic charge-discharge |
| GNPs | Graphene nanoplatelets |
| GO | Graphene oxide |
| HCN | Holey carbon nanolayers |
| HRTEM | High-resolution transmission electron microscopy |
| HS | Hybrid supercapacitors |
| LFP | Lithium iron phosphate |
| LIBs | Lithium-ion batteries |
| $LiCoO_2$ | Lithium cobalt oxide |
| $LiFePO_4$ | Lithium iron phosphate |
| $LiMn_2O_4$ | Lithium manganese oxide |
| MES | Mesoporous carbon |
| $MnO_2$ | Manganese dioxide |

| | |
|---|---|
| Ni-Cd | Nickel-cadmium |
| NiO | Nickel oxide |
| $R_{ct}$ | Charge transfer resistance |
| $R_s$ | Solution resistance |
| rGO | Reduced graphene oxide |
| $RuO_2$ | Ruthenium (IV) oxide |
| SAED | Selected area electron diffraction |
| Si NPs | Silicon nanoparticles |
| TiN | Titanium nitride |
| XPS | X-ray photoelectron spectroscopy |
| XRD | X-ray Diffraction |
| $Z_w$ | Warburg impedance |

# CHAPTER 1

# INTRODUCTION

## 1.1 Research background

Supercapacitors and secondary batteries are the two main technologies used to store energy electrochemically. However, none of them is able to satisfy the growing demands for electrical energy storage (EES) system with high energy, high power, and long life-cycle in a single device (Qu et al., 2019). The emergence of hybrid supercapacitors (HS) has become recognizable among researchers. HS combines the underlying structures of both lithium-ion batteries (LIB) and electrical double layer capacitors (EDLC) into one physical unit. These hybrid components are not just a simple packaging of a distinct battery and supercapacitor pair in a common housing. Instead, they are energy sources that merge the chemistry of a battery with the physics of a supercapacitor into a single design. As a result, these hybrid devices offer a synergic advantage from both technologies (higher energy density from the battery and high-power capability, long life-cycle from the supercapacitor), and eliminate their drawbacks (Peng et al., 2018; Ding et al., 2018; Aravindan & Lee, 2018).

Like many other energy storage technologies, HS has four components: (1) anode, (2) cathode, (3) electrolyte, and (4) separator. The HS uses a battery type cathode and capacitive type anode materials in an asymmetrical cell configuration. The charge storage mechanism in HS is based on the formation of an electrical double layer at the capacitive type electrode and ion intercalation/deintercalation at the battery type electrode (Li et al., 2018). Lithium iron phosphate (LFP) has been recognized as a potential candidate to replace lithium cobalt oxide and lithium manganese oxide as cathode materials in LIBs due to its high theoretical specific capacity of 170 mA h $g^{-1}$, low toxicity, great cycle stability, and excellent chemical and thermal stability (Hsieh et al., 2021). Moreover, Fe is an abundant element, which reduces the fabrication cost, and the phosphate anion is a notably robust anion to make a safe LIB with a high theoretical capacity for high energy density and rate capability (Tron et al., 2017).

The electrical conductivity of LFP composites are enhanced by means of incorporation of carbon materials (Rueda-Garcia et al., 2018), metal or metal oxide (Cui et al., 2010), and Li- or Fe-site ion doping (Liu et al., 2020; Wang et al., 2012). Carbon-based materials such as graphene, activated carbon, and carbon nanotube coating on the LFP surface are found to be very effective ways to improve the electronic conductivity of LFP. As a superior two-dimensional conductive material, graphene with a high specific surface area, high specific capacitance, and excellent ion conductivity has been widely employed in energy storage devices (Olabi et al., 2021). Additionally, the surface modification with silicon (Si) is introduced to further enhance the capacity as well as the surface stability of LFP material by preventing the side reaction on the surface of LFP material in aqueous electrolytes (Lin et al., 2013). The silicon-oxygen bond that is created between the Si element and the oxygen element in LFP materials has strengthened the composites, preventing them from easily collapsing during charging/discharging processes. Si is also highly capable of forming a silicon-carbon bond with graphene, enabling the produce impactful and effective LFP composites. Therefore, such a combination of special structures and materials (graphene and Si) could mitigate the detrimental behaviour of the LFP cathode and be a promising candidate for advanced HS. Many studies have been conducted on Li-based EES devices using carbon-based cathode materials. For instance, $LiFePO_4$ and $LiMn_2O_4$ composites were fabricated with mesoporous carbon (MES) for HS application (Ortega et al., 2018; dos Santos Junior et al., 2019). The mesoporous surface area of MES had enabled the insertion of $Li^+$ from the electrolyte into the electrodes and facilitated the ion transportation. In the other work, a hybrid Li-ion capacitor using carbon-coated LFP (LFP@C) composite, where a uniform carbon layer was formed on the LFP composite, had improved the LFP conductivity and reduced the diffusion resistance of $Li^+$ in the system (Gao et al., 2019). A Li-based symmetric supercapacitor based on $LiMnO_3$/carbon fabric cloth (CFF) electrodes was able to assist the electrochemical performance by shortening the ion transport path for fast redox reactions by taking into account the high specific surface area of CFF (Xu et al., 2018). A Li-ion supercapacitor based on the C-$LiFePO_4$/TiN cathode, in which the carbon-coated LFP that was supported on a titanium nitride (TiN) substrate, was produced to intensify the electronic conductivity (Xie et al., 2015). Meanwhile, holey carbon nanolayers (HCN) with a porous structure, when utilized as cathode and anode electrodes, were able to achieve interlayer spacing and electronic conductivity for Li-ion diffusion as demonstrated in a Li-ion HS (Li et al., 2018; Zhao et al., 2019). Other than that, the incorporation of carbon nanotubes (CNTs) and graphene into the $LiMn_2O_4$ composites ($LiMn_2O_4$@CNTs@graphene) in HS application were able to facilitate the charge storage process and fasten the kinetics of Li-ion (Chen et al., 2019). Overall, the HS systems are mostly integrated with the goal of higher electrochemical performance based on carbon-based materials, and to enhance the specific capacitance and energy density.

Based on the aforementioned advantages of the hybrid cathode material, LFP modified silicon and graphene derivatives (LFP/Si/graphene) are synthesized via two-step synthesis of hydrothermal and microwave-assisted synthesis. Graphene nanoplatelets (GNPs) and reduced graphene oxide (rGO) play a role as electronic conductivity enhancers for LFP and a binder for Si in the framework structure of rGO film. This interaction forms a chemical bond between Si and the oxygen functional groups of GO, which can stabilize Si in the composite structure. Different loadings of Si are introduced to LFP to evaluate the optimum amount of Si in the composite materials for obtaining excellent capacity and energy density of the HS device. Aqueous electrolytes with super stability are inexpensive and can guarantee the safety of the EES device (Pan et al., 2020). The electrochemical performance of fabricated electrode is evaluated in 1 M $Li_2SO_4$ aqueous electrolyte which thoroughly deaerated with high-purified argon (Ar) gas prior to electrochemical testing. Ar blanketing in the electrolyte intensified the electrochemical performance by 60% as compared to the Ar-free electrolyte. The presence of Ar in electrolyte has prevented the oxygen interference that could hinder the electrochemical performance. Besides, laboratory testing of HS cell is performed in Swagelok and roll-type cell configuration, where two electrodes and electrolyte-saturated separators are placed in a tightly packed cell assembly. A comparison of HS performance across different cell format, a roll-type cell was investigated and exhibit a slight enhancement in the specific capacitance and cycle stability over 5200 cycles. Therefore, both cell configurations depicted a comparable HS performance and the cell assemblies are performed in a close-to-current commercial format, which enables a realistic and consistent evaluation of HS cell. This aqueous-based LFP/Si/graphene HS is illustrated in **Figure 1.1.**

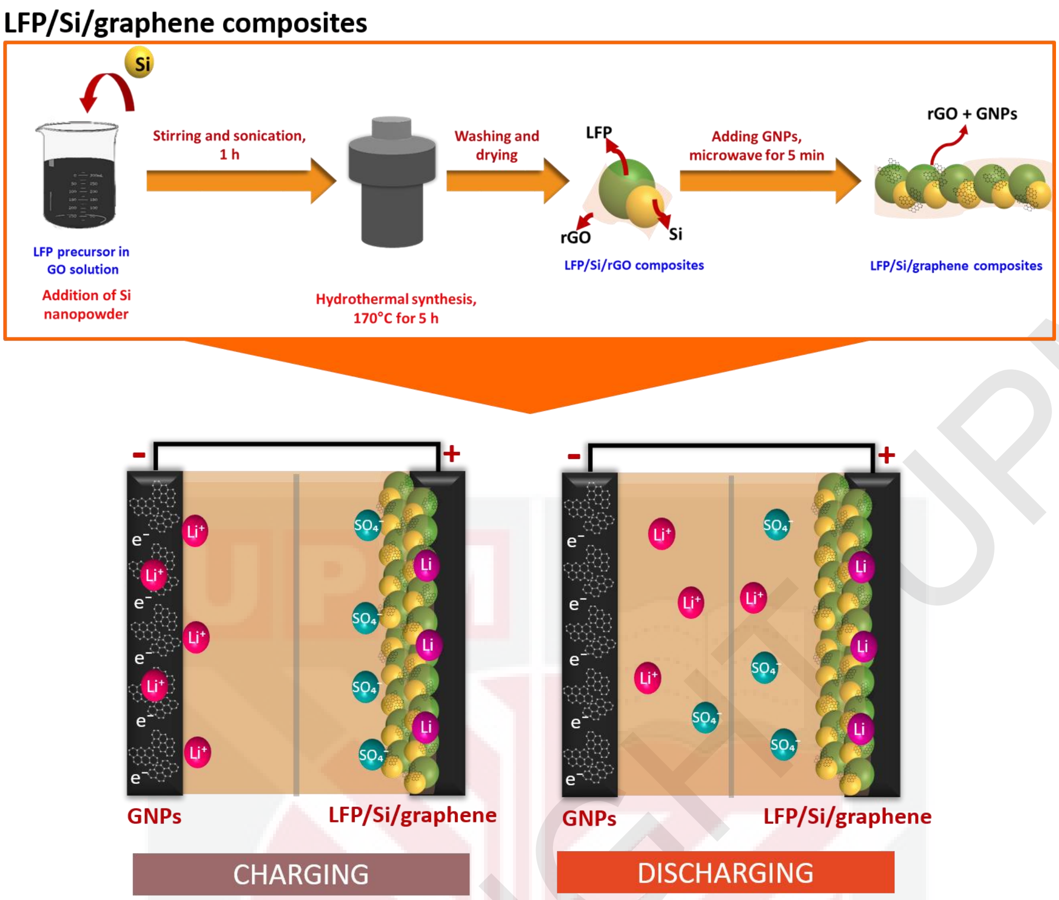


**Figure 1.1: Schematic diagram of aqueous-based LFP/Si/graphene HS synthesis.**

## 1.2 Problem statements

Despite offering the benefits, it is important to acknowledge the significant drawbacks of bare LFP materials that are negatively impacted the electrochemical performance, including poor electrical conductivity of bare LFP (~ $10^{-9}$ to $10^{-11}$ S $cm^{-1}$), low diffusion coefficient of lithium-ion ($10^{-11}$ to $10^{-13}$ $cm^2$ $s^{-1}$), and surface instability issues of LFP composites. These limitations of bare LFP have subsequently caused the capacity fading and poor rate capability, posing a bottleneck for the energy storage application (Yang et al., 2016; Satyavani et al., 2016).

Fortunately, graphene has featured with its exceptional properties to overcome the drawbacks of bare LFP, allowing the materials to accommodate the volume changes during charging and discharging cycles, protect the electrode integrity during longer cycling process, and facilitate an efficient electron transfer (Xu et al., 2012; Zhang et al., 2021). The incorporation of graphene into LFP composites has provided a conductive network in LFP structures, proving the enhanced electronic conductivity that positively addressed the limitations of bare LFP materials (Xu et al., 2012; Zhang et al., 2021; Gao et al., 2019; Sanchez et al., 2021). However, while improving the electronic conductivity, graphene has a low contribution in intensifying the specific capacity or energy density of energy storage application. There are other alternative strategies for these challenges, such as doping LFP with additional metal to increase the discharge voltage plateau (Zhang et al., 2012) or reducing the particle size of LFP to shorten the lithium-ion diffusion pathways (Tian et al., 2017). The increase in voltage plateau based on the doping process has benefited the electronic structures and redox behaviours, enhancing the electronic conductivity of LFP structures and mitigating the capacity fade during cycling activities (Zhang et al., 2012). Meanwhile, the smaller particle sizes of LFP are capable of reducing the diffusion distance of $Li^+$ to travel between the electrode and electrolyte, exhibiting a faster charge/discharge rate and improving the electrochemical kinetics (Tian et al., 2017). Nevertheless, these approaches are only applicable for the improvement of specific capacity and energy density, leading to deterioration of material stability and reduction of cell lifespan. Thus, Si are employed as the promising element to cure the surface instability issues and prevent the side reaction on LFP in aqueous electrolytes, effectively mitigating the capacity fading and maximizing the cell lifespan by promoting a stable interface and more efficient Li-ion transportation between electrodes.

Furthermore, organic electrolytes are extensively utilized in lithium-ion-based EES devices due to their prominent power density and compatibility with electrodes, but their downsides are inevitable. The downsides of these electrolytes, such as low safety, high volatility and flammability, high sensitivity to moisture, and high cost, have constrained and hindered their further widespread applications and raised the concerns about environmental impact. These electrolyte limitations have increased the safety risks and reduced the reliability of energy storage application. To address these aspects, the application of aqueous electrolytes in EES devices has been introduced as a potential candidate to intensify the electrochemical performance with more environmental friendly approach.

## 1.3 Research objectives

The objective of this study are:

1. To synthesize the lithium iron phosphate (LFP) composites with the association of silicon nanoparticles (Si NPs) and graphene derivatives for cathode active material of Li-based HS.
2. To fabricate and analyze the electrochemical performance of aqueous HS based on LFP/Si/graphene composite.

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