# **WORK PERMIT**

#### Department of Chemical and Biological Engineering

#### 化學及生物工程學系

Project Title: MoS2/S Composite with Nitrogen-Decorated

Porous Carbon from MOF as Cathode for

High Performance Lithium Sulfur Battery

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Signature of Approval:

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Work Plan No.: \_\_\_\_\_17046\_\_\_\_\_

Date of Approval:

Date of Revalidation: N/A

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### The Hong Kong University of Science & Technology

## **Department of Environmental Engineering**

#### **Work Plan**

Work plan Number:

17046

**Project Title:** 

MoS<sub>2</sub>/S Composite with Nitrogen-Decorated Porous

Carbon from MOF as Cathode for High Performance

Lithium Sulfur Battery

Name of Researcher:

**GU Shaonan** 

Name of Supervisors:

Prof. CHEN Guohua

Research Area:

'Room 7107

**Proposed Location:** 

Room 7107

Start date:

10/07/2017

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#### 1. General Information

Name of Researcher: GU Shaonan

Name of Supervisors: Prof. CHEN Guohua

Work plan No.: 17046

Project title: MoS<sub>2</sub>/S Composite with Nitrogen-Decorated Porous Carbon from MOF

as Cathode for High Performance Lithium Sulfur Battery

Research area: Synthesis of composite nanomaterials, Lithium battery

Proposed location: Room 7107

Start date: 10/07/2017

#### 2. Experiment/Project Description

The energy storage market has increased dramatically over the last few decades due to the demand for energy storage systems expanding beyond portable electronic devices to electric vehicles and smart grids. Among the various alternative energy storage systems, one of the most prominent is a lithium-sulfur (Li-S) battery that utilizes sulfur as a cathode material by converting it into lithium sulfide. In doing so, it provides a high theoretical capacity of 1675 mA h g<sup>-1</sup>, which corresponds to a high theoretical specific energy and volumetric energy density of ~2835 W h L<sup>-1</sup> and ~2572 W h kg<sup>-1</sup>, respectively, at an open-circuit voltage (OCV) of ~2.18 V. The chemistry of a Li-S battery is fundamentally different from the intercalation process that occurs in conventional Li-ion batteries, which offers both significant advantages but also some rather critical challenges that still need to be addressed. The main drawbacks that Li-S battery is suffering include: low conductivity of sulfur and lithium sulfide; large volumetric expansion of sulfur upon lithiation and dissolution of intermediate lithium polysulfides into the electrolyte. All these drawbacks can induce the low Coulombic efficiency and poor battery performance. To overcome these issues, my research project is to synthesize a desirable composite for high performance Li-S battery using as cathode. My target is to preparation a novel cathode material MoS<sub>2</sub>/S@NPC (N decorated porous carbon) with high energy density and stable cycling performance.

#### 2.1 Objectives

Major objectives of this study is: 1) to synthesize a series proportion of MoS<sub>2</sub>/S@NPC composites. 2) to characterize the structure and electrochemical properties of as prepared cathode material. 3) to evaluate the electrochemical performance of the assembled coin-type cells and clarify the mechanism of the enhancement in battery performance.

#### 2.2 Experiment

Synthesis of ZIF-8, and N-decorated porous carbon

2-methylimidazole (2-MeIM, 3.284g, 40 mmol) is dissolved in 100 mL of methanol to form a transparent solution A. Then, a solution of Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (2.975 g, 10 mmol, 100 mL) in methanol is added to form a homogeneous system. Under vigorous stirring at room temperature, the transparent solution turn turbid gradually.

After stirring for 1 h, the solution is incubated for 48 h. After centrifugation, washing with methanol several times, and drying at 60 °C overnight, the white ZIF-8 products will be obtained.

The as-prepared ZIF-8 sample (200 mg) is dispersed in excess of furfuryl alcohol (FA, 5 mL) and NH<sub>4</sub>OH (0.5 mL) followed by stirred for 12 h. After careful filtration and washing with ethanol, the FA-NH<sub>4</sub>OH/ZIF-8 composite is charged into a tube furnace under a Ar flow, heat-treated at 80 °C for 24 h and then at 150 °C for 7 h for FA polymerization, and finally calcined at 1000 °C for 8 h with a heating ramp of 10 °C min<sup>-1</sup>.

(Alternative procedure: Copper acetate monohydrate (8 mg, 0.04 mmol) and 2,3,6,7,10,11-hexahydroxytriphenylene (HHTP, 6.5 mg, 0.02 mmol), are dispersed in 1 mL solvent mixture of water/methanol (v: v = 1:1) under sonication for 10 min in a 20 mL glass vial. The reaction mixture is heated in an isothermal oven at 85 °C for 12 h resulting in dark blue crystals. The reaction mixture is allowed to cool naturally to room temperature and the crystals are washed with deionized water and then retained for use)

#### Synthesis of MoS<sub>2</sub>/S@NPC

Sulfur and N decorated porous carbon are thoroughly mixed with a mass ratio of Ms:Mc = 5:1. After that, the mixture is thermally treated under  $Ar/N_2$  at 155 °C for 6 h. During the heating treatment, a slow heating rate of 0.5 °C min<sup>-1</sup> above 115 °C (the melting point of sulfur) helps the sufficient infiltration of sulfur into carbon matrix.

123.6 mg (0.1 mmol) Ammonium molybdate tetrahydrate is dissolved in 10 ml ethanol and 600 mg S@NPC powders is added and dispersed under supersonic. Then the suspension is stirred during the evaporation on a heating jacket. Afterwards, the sediment is charged into a tube furnace under an Ar flow, heat-treated at 350 °C for 2 h.

(Alternative procedure: 123.6 mg (0.1 mmol) Ammonium molybdate tetrahydrate is dissolved in 55 ml distilled water and 600 mg S@NPC powders is added and dispersed under supersonic. After 5 mL of hydrazine solution ( $N_2H_4H_2O$ , 35%) as reducing agent is injected, the mixture solution transferred into a 100 ml Teflon-lined stainless steel autoclave and heated at 160 °C for 12 h. After filtering, black powders are collected, dried at 60 °C for 5 h and then retained for use)

# 3. Equipment List

Equipment	Location
Centrifuge	6/F CBME Corridor
Drying oven	7/F ENV Corridor
Vacuum tube furnace	7/F ENV Corridor
Magnetic stirrer	Room 7107
Electronic balance	Room 7107
Autoclave	Room 7107
Powder-pressing machine	Room 7107
Battery testing channel	Room 7107
Argon glove box	Room 7107
Nitrogen glove box	7/F ENV Corridor
Capping machine	7/F ENV Corridor
Ultrasonicator	7/F ENV Corridor
Tube furnace	7/F ENV Corridor
Muffle furnace	7/F ENV Corridor
Thermogravimetric analyzer (TGA)	Room 7101
BET	Room 7106
FT-IR	Room 2153
Raman Spectra	Room 2153

#### 4. Experimental procedure

#### 4.1 Fabricate procedures

This experiment is consist of established experimental procedures, and references are listed at the end of this chapter.

#### 4.1.1 Material preparation

- (1) 2-methylimidazole (2-MeIM, 3.284g, 40 mmol) dissolves in 100 mL of methanol. Then, a solution of Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (2.975 g, 10 mmol, 100 mL) in methanol is added in. (Finished on bench top in 7107)
- (2) Under vigorous stirring at room temperature, the above solution is incubated for 48 h. (Finished in fume-hood with a magnetic stirrer in 7107)
- (3) After centrifugation, washing with methanol several times, and drying at 60 °C overnight, the white ZIF-8 products will be obtained. (Finished with centrifuge and dry oven)
- (4) ZIF-8 sample (200 mg) disperse in excess of furfuryl alcohol (FA, 5 mL) and NH<sub>4</sub>OH (0.5 mL) followed by stirred for 12 h. (*Finished in fume-hood with a magnetic stirrer in 7107*)
- (5) After filtration and washing with ethanol, the FA-NH<sub>4</sub>OH/ZIF-8 composite is charged into a tube furnace under a Ar flow, heat-treated at 80 °C for 24 h and then at 150 °C for 7 h, and finally calcined at 1000 °C for 8 h with a heating ramp of 10 °C min<sup>-1</sup>. (Finished with centrifuge and tube furnace with Ar flow in 7/F ENV Corridor)

(Alternative procedure 1)  $Cu(Ac)_2 \cdot H_2O$  (8 mg, 0.04 mmol) and HHTP (6.5 mg, 0.02 mmol), are dispersed in 1 mL solvent mixture of water/methanol (v: v = 1:1) under sonication for 10 min in a 20 mL glass vial. (Finished on bench top with a magnetic stirrer in 7107)

(Alternative procedure 1') Then the mixture will be heated in oven at 85 °C for 12 h followed by washed with deionized water and then retained for use. (Finished on bench top in 7107 and dry oven in 7/F ENV Corridor)

(6) The mixture of S and NPC is thermally treated under Ar flow at 155 °C for 6 h. (Finished with tube furnace uder Ar flow in 7/F ENV Corridor)

- (7) Ammonium molybdate tetrahydrate is dissolved in 10 ml ethanol and 600 mg S@NPC powders is added and dispersed under supersonic. Then the suspension is stirred during the evaporation. (*Finished in fume-hood in 7107*)
- (8) Afterwards, the sediment is charged into a tube furnace under Ar flow, heat-treated at 350 °C for 2 h. (Finished with tube furnace under Ar flow in 7/F ENV Corridor)

(Alternative procedure 2) Ammonium molybdate tetrahydrate is dissolved in 55 ml DI water and 600 mg S@NPC powders and 5 mL of hydrazine solution ( $N_2H_4$   $H_2O$ , 35%) are added and dispersed under supersonic. Then the suspension is transferred into a 100 ml Teflon-lined stainless steel autoclave and heated at 160 °C for 12 h. (Finished in fume hood in 7107 and dry oven in 7/F ENV Corridor)

(9) Washing the final product with carbon disulfide (CS<sub>2</sub>, 5 mL) three times to remove redundant sulfur on the surface of as-prepared composite. (Finished in fume-hood in 7107)

#### 4.1.2 Cell assembly

- (1) Certain ratio of cathode materials (0.7g), acetylene black (0.2g), PVDF (0.1g) and NMP (2 g) are mixed by grinding. (Finished in milling machine in 7107)
- (2) The composite will be dried at 140 °C for 2 h. (Finished in oven in 7/F ENV Corridor)
- (3) The coin cell will be fabricated with pellet, collector, electrolyte (DOL/DME/LiTFSI/LiNO<sub>3</sub>) and lithium foil, in argon-filled glove box. (*Finished in Glovebox in 7107*)

#### References

- Seh Z W, Sun Y, Zhang Q, et al. Chemical Society Reviews, 2016, 45(20): 5605-5634.
- 2. Wang S, Zhao Z, Xu H, et al. *Electrochimica Acta*, 2015, 173: 282-289.
- 3. Aijaz A, Fujiwara N, Xu Q. Journal of the American Chemical Society, 2014, 136(19): 6790-6793.
- 4. Zhou J, Li R, Fan X, et al. Energy & Environmental Science, 2014, 7(8): 2715-2724.
- 5. Wang D, Pan Z, Wu Z, et al. Journal of Power Sources, 2014, 264: 229-234.

6. Jiang H L, Liu B, Lan Y Q, et al. *Journal of the American Chemical Society*, 2011, 133(31): 11854-11857.

#### 4.2 Electrochemical performance evaluation

Both electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) measurements will be taken at ambient temperature using solid-state electrochemical cells assemblies by sandwich electrolyte membrane between two planar electrodes.

#### 5. Procedure Template

Experimental	Experimental	Scale	Location	Method
	Procedure			
Procedure No.	Description	(Mass/Volume,	(Bench top,	New or Existing
		etc.)	etc.)	
4.1.1(1)	Dissolve	(1) 3.284 g	Bench top in	Existing
	(1) 2-MeIM,	(2) 2.975 g	7107	
	$(2)Zn(NO_3)_2, (3)$	(3) 200 mL		
	methanol		•	
4.1.1 (2)	Vigorous stirring	48 h	Bench top in	Existing
			7107	
4.1.1 (3)	Centrifugation,	12 h	Centrifuge and	Existing
	washing and		dry oven in 7/F	
	drying at 60 °C		ENV Corridor	
4.1.1 (4)	Disperse	(4) 5 mL	Bench top in	Existing
	Sample ZIF-8 in	(5) 0.5 mL	7107	
	(4) Furfuryl			
	alcohol			
	(5) NH <sub>4</sub> OH			
4.1.1 (5)	Heat obtained	80 °C for 24 h,	Tube furnace	Existing
	sample above	and then	with Ar flow in	
	under Ar flow in	150 °C for 7 h,	7/F ENV	
	a tube furnace	1000 °C for 8 h	Corridor	

4.1.1 (A1)	Dissolve and	(20) 8 mg	Bench top in	Existing
	stirring (20)	(21) 6.5 mg	7107	
	Cu(Ac) <sub>2</sub> and (21)			
	ННТР			
4.1.1 ( <i>A1</i> ')	Obtained powder	85 °C for 12 h	Dry oven in 7/F	Existing
	from last step is		ENV Corridor	
	heat in oven			
4.1.1 (6)	The mixture of	(6) 500 mg	Tube furnace	Existing
	(6) Sulfur and		with Ar flow in	
	NPC is thermally		7/F ENV	
	treated		Corridor	
4.1.1 (7)	Dissolve and	(7) 123.6 mg	Bench top in	Existing
	dispersion	as-prepared	7107	
	(7)	S@NPC 600		
	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> .4	mg		
	H <sub>2</sub> O and as-			
	prepared			
	S@NPC			
4.1.1 (8)	Heat the	350 °C for 2 h	Tube furnace	Existing
	sediment under		with Ar flow in	1
	Ar flow in a tube		7/F ENV	
	furnace		Corridor	
4.1.1 ( <i>A2</i> )	5 mL (19) N <sub>2</sub> H <sub>4</sub>	160 °C for 12 h	Dry oven in 7/F	Existing
	H <sub>2</sub> O, 35%) is		ENV Corridor	
	added in 4.1.1			
	(7) and			
	transferred to			
	autoclave for			
	heating in oven			
4.1.1 (9)	Washing the	5 mL for three	Fume-hood in	Existing
	composite with	times	7107	
	(22) CS <sub>2</sub>			
A1 and A2 are abb	reviation for the alt	ternative procedure	es listed in Section	4.1.1
4.1.2 (1)	Mix (8) PVDF in	(8) 0.1 g	Fume hood	Existing
	1	_		I -
(1)	(9) NMP under	(9) 2 g	with a magnetic	

	Mill (8) PVDF,	(10) AC 0.2 g	Milling	Existing
	(9)NMP, (10)	MoS <sub>2</sub> /S@NPC	machine in	
	acetylene black	0.7 g	7107	
	and cathode			
	materials to get			
	electrode			
4.1.2 (2)	Dry the	140 °C for 2 h	Oven in 7/F	Existing
	composite		ENV Corridor	
	obtained above			
4.1.2 (3)	Vacuum-dried	Electrolyte	Oven in 7/F	Existing
	electrolyte/electr	(11) DOL/(12)	ENV Corridor	
	ode to remove	DME/(13)		
	water before put	LiTFSI/(18)		
	into glovebox	LiNO <sub>3</sub> < 20 mg		
	Press	(14) Aluminum	Glovebox in	Existing
	electrode/electrol	foil/electrode:	7107	
	yte/electrode	/(8) PVDF/		
	under certain	(11) AC<10	,	
	pressure using a	mg/(15) Li Foil		
	coin cell to sea	Electrolyte	Glovebox in	Existing
		film; two	7107	
		electrodes and		
·		coin cell		

# 6. HAZOP Template

A 04:-:4-:		Hazard and Operability Analysis	lity Analysi	S			
ACHVILY:							
No	Hazard	Hazard Effect	Severity	Probability	Risk	Minimize Risk By	Residual Risk
4.1.1 (1) Conta	Contact with Zn(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O	Skin and eye irritation. Cause fire	M	Z	Z	Wear protective gloves,	J
and 2 flamr	and Z-MelM. Methanol is flammable					goggles, face shield and lab coats. Keep away	
						from fire/heating source	
4.1.1 (2) The s	The same with (1)	The same with (1)	M	$\mathbb{Z}$	Σ	Keep the incubation in	J
						fume hood	
4.1.1 (3) Hot s	Hot surface of the Oven	Skin burn if touched	Γ	П	П	Set a warning sign and	L
						operate with protective	
						gloves	
4.1.1 (4) Cont	Contact with Furfuryl alcohol	Skin and eye irritation. Cause fire	Γ	×	$\boxtimes$	Wear protective gloves,	Н
and D	and NH4OH. Furfuryl alcohol					goggles, and lab coats.	
is fla	is flammable					Keep away from	
						fire/heating source	
4.1.1 (5) Wron	Wrongly programed tube	Damage of the tube furnace inside	$\mathbb{Z}$	J	Z	Set up the tube furnace	Г
furna	furnace setting					carefully and check the	
						temperature within 2 h	
4.1.1 ( <i>AI</i> Cont	Contact or inhale HHTP, and	Causes irritation of the lungs and	$\mathbb{Z}$	×	Σ	Wear protective gloves,	Г
and AI? hot so	hot surface of the Oven	respiratory system. Irritating to eyes				goggles, and lab coats.	
		and skin on contact. Skin burn if				Keep away from	
		touched				fire/heating source	

4.1.1 (6)	Powder (Sulfur and NPC) and	Causing respiratory tract, and the same	M	M	$\mathbb{Z}$	Using protecting mask	Г
	the same with (5)	with (5)				and gloves. Conduct this	
						step in fume hood, and	
						the same with (5)	
4.1.1 (7)	Ethanol is flammable	Cause fire	M	T	$\mathbb{M}$	Keep away from	Г
						fire/heating source	
4.1.1 (8)	The same with (5)	The same with (5)	M	Γ	$\mathbb{Z}$	The same with (5)	Γ
4.1.1 (42)	Contact with N <sub>2</sub> H <sub>4</sub> H <sub>2</sub> O, and	Causes severe skin burns and eye	M	Γ	$\mathbb{Z}$	Using protecting mask	Γ
	the same with (3)	damage. May cause an allergic skin				and gloves. Conduct this	
		reaction and the same with (3)				step in the fume hood,	
						and the same with (3)	
4.1.2 (9)	Contact with CS <sub>2</sub> . CS <sub>2</sub> is	Causes skin irritation. Causes serious	M	M	$\boxtimes$	Wear protective gloves,	Γ
	flammable	eye irritation. Cause fire				goggles, face shield and	
						lab coats. Keep away	
						from fire/heating source	
4.1.2	The same with (3)	The same with (3)	Н	J	Н	The same with (3)	-
(1)~(3)	Contact or inhale PVDF,	Causing respiratory tract, skin and eye				Operate these steps in	M
	NMP, AB, LiTFSI, DOL,	irritation				glovebox	
	DME and powders						
FINAL AS	SESSMENT: With proper PPE eq	FINAL ASSESSMENT: With proper PPE equipped and careful operation of furnace, 4.1&4.2 can be done	&4.2 can b	e done		OVERALL RISK:	T
safely.							

Remark: Severity-L=Low (Minor injuries, first aid); M=Medium (Hospitalization, medical leave); H=High (Serious injuries, fatality)

Probability-L=Low (Unlikely); M=Medium (Possible); H=High (Very Likely)

Note: Severity x Probability = Risk [eg. LxL=L; LxM=M; LxH=H; HxM=H; the product follows the higher severity or probability]

Higher Risk requires extensive risk minimization procedures

#### 7. Operating Conditions

Preparation step operate in the fuming cupboard under room temperature. And all the chemicals will mix in the beaker with natural pH value. The concentration of each solution has been listed above in the section of 2.2. Dry steps (autoclave reaction) are taken in the oven at certain temperature (60°C, 85 °C, 160°C) and standard pressure. Calcination steps are processed at 80°C, 150°C, 350°C and 1000°C respectively under Ar flow.

#### 8. Services List

Electric power: 220V

Water: distilled water, tap water, DDI water

Gases: Ar, air

Face mask to prevent breath in the fine powder

Eye shields

Full-face particle respirator type

Gloves

Against chemicals coat

#### 9. Chemical list

No.	Chemical	Purity %	Quantity	per MSDS attached
			Experiment	
(1)	2-methylimidazole	99%	3.284 g	Yes
(2)	Zn(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O	>99%	2.975 g	Yes
(3)	Methanol	/	/	Yes
(4)	Furfuryl alcohol	98%	5 mL	Yes
(5)	NH <sub>4</sub> OH	25%	/	Yes
(6)	Sulfur	99.98%	5 g	Yes
(7)	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> .4H <sub>2</sub> O	99.98%	0.2 g	Yes

(8)	PVDF	/	0.1g	/
(9)	NMP	HPLC grade	2 g	Yes
(10)	Acetylene black	/	0.2 g	
(11)	DOL	/	Electrolyte 2g	
(12)	DME	99.95%		Yes
(13)	LiTFSI	99.99%		
(14)	Al foil	/	5 pieces	/
(15)	Li foil	/	5 pieces	/
(16)	Ar	/	/	/
(17)	Alcohol	/	/	Yes
(18)	LiNO <sub>3</sub>	99.99%	0.5 g	Yes
(19)	N <sub>2</sub> H <sub>4</sub> H <sub>2</sub> O	35%	10 mL	Yes
(20)	Cu(Ac) <sub>2</sub>	99.99%	0.5 g	Yes
(21)	ННТР	95%	0.2 g	Yes
(22)	CS <sub>2</sub>	99.99%	30 mL	Yes

## 10. Biological Agent list

None

# 11. Summary of Relevant Hazards and Incompatibilities

Chemical	Hazards	Incompatibilities
2-Methylimidazole	Be harmful if swallowed. Causes skin	Strong oxidizing agents,
	burns and eye damage when contact with	acids, Acid chlorides, Acid
	substance. May damage fertility or the	anhydrides
	unborn child.	
Zinc nitrate	May intensify fire; oxidizer. Causes skin	Powdered metals, Cyanides,
hexahydrate	and serious eye irritation when contacted.	Sodium hypophosphite,
		Stannous chloride,
		Thiocyanates, Strong
	U	reducing agents.

LiNO <sub>3</sub>	Inhalation, ingestion or contact (skin,	Strong reducing agents,
	eyes) with vapors or substance may cause	combustibles, organic
	severe injury, burns or death. Fire may	materials.
		materiais.
	produce irritating, corrosive and/or toxic	
<u></u>	gases.	
Ethanol	Highly flammable liquid and vapor.	Alkali metals, Oxidizing
Methanol	Highly flammable liquid and vanou	agents, Peroxides
Methanol	Highly flammable liquid and vapor.	Acid chlorides, Acid
	Toxic if inhaled and contacted with skin.	anhydrides, Oxidizing agents,
	Causes damage to organs.	Alkali metals, Reducing
		agents, Acids
Acetylene black	Irritant and highly flammable.	Keep/Store away from
		oxidizing agents, acids,
		halogens.
Furfuryl alcohol	May cause damage to organs (Nose)	Air sensitive. Do not store
	through prolonged or repeated exposure	near acids., Oxygen, Strong
	if inhaled. Toxic if swallowed or in	oxidizing agents
	contact with skin. May cause respiratory	
	irritation.	
NH <sub>4</sub> OH	Causes severe skin burns and eye	Oxidizing agents, Oxygen,
	damage. Very toxic to aquatic life. Avoid	Copper, Organic materials,
	release to the environment.	Zinc
PVDF	Not a dangerous substance	Strong oxidizing agents,
		Titanium/titanium oxides.
1,3-Dioxolane	Highly flammable liquid and vapor. Keep	Strong oxidizing agents
(DOL)	away from heat, hot surfaces, sparks,	
	open flames and other ignition sources.	
1,2-	Highly flammable liquid and vapor. Keep	Oxidizing agents, Strong
Dimethoxyethane	away from heat, hot surfaces, sparks,	acids
(DME)	open flames and other ignition sources.	
LiTFSI	Toxic if swallowed or in contact with	Strong oxidizing agents
	skin. Causes severe skin burns and eye	
	damage. May cause damage to organs	
	through prolonged or repeated exposure.	
	Harmful to aquatic life with long lasting	
	effects.	
	Officers.	

Lithium Metal	Highly flammable and Corrosive.	Forms shock-sensitive
		mixtures with certain other
		materials, e.g. iron and iron
		salts, heavy metals,
		phosphorus, sulphur
		compounds, oxygen and
		nickel. Do not store near
		acids, metals.
NMP	Toxicological information: Danger. May	Strong acids, Strong oxidizing
	cause skin irritation, serious eye	agents.
	irritation, and respiratory irritation.	
	Hazardous Decomposition Products:	
	Stable under recommended storage	
	conditions.	
N <sub>2</sub> H <sub>4</sub> H <sub>2</sub> O	Toxic if swallowed or in contact with	Oxidizing agents, Oxygen,
	skin. Causes severe skin burns and eye	Copper, Organic materials,
	damage. Fatal if inhaled. Very toxic to	Zinc
	aquatic life with long lasting effects.	
ННТР	Inhalation causes irritation of the lungs	Strong acids, Strong oxidizing
	and respiratory system. Irritating to eyes	agents.
	and skin on contact. Keep away from	
i	fire/heating source	
CS <sub>2</sub>	Highly flammable liquid and vapor.	Alkali metals, Zinc, Amines,
	Causes skin irritation. Causes serious eye	Azides, Oxidizing agents
-	irritation.	

#### 12. Waste List

Liquid waste generated in the synthesis and cell fabrication processing is mainly solvent, such as methanol, ethanol, NMP with low concentration of Zn and Li ions and polymer like PVDF-HPF.

All liquid waste will be discharged to an appropriate closed container. Data log sheet, which details solution volume and composition, will be submitted with container. (Maximum 10 L per month, average 2.5 L per month)

Residual resins from electrochemical performance studies will be disposed as municipal waste. Data log sheet contains information regarding the nature of wastes will be provided. (Maximum 30 g per month, average 10 g per month)

Used consumables such as disposable gloves, paper towel, etc. will be disposed into the rubbish bin with plastic waste bag in Room 7107. The plastic bag will be sealed carefully before leaving the room. Contact technical staff to arrange for special removal.

#### **Chemical Waste Log Sheet**

<200 mL	Non-halogenated Solvents	Non-halogenated
	Colvente	
	Solvents	Solvents
<50 mL	Non-halogenated	Non-halogenated
	Solvents	Solvents
<20 mL	Non-halogenated	Non-halogenated
	Solvents	Solvents
<50 mL	Non-halogenated	Non-halogenated
	Solvents	Solvents
<20 mL	Non-halogenated	Non-halogenated
	Solvents	Solvents
<5 g	Halogenated	Halogenated solvent
	Solvents	container
<10 g	Standard	Standard chemical
	chemical waste	waste containers
<5 pieces	Standard	Standard chemical
	chemical waste	waste containers
<5 pieces	Lithium foils	Metal solution
		container*
	<20 mL <50 mL <20 mL <10 g <5 pieces	Solvents  Non-halogenated Solvents  Non-halogenated Solvents  Non-halogenated Solvents  Solvents  Halogenated Solvents  Chemical waste

<sup>\*</sup> For small quantities (several grams) of Li foil, they can be allowed to react in isopropyl alcohol in fume hood then the solution can be transferred to alkali or metal solution. Li should be fully reacted before discard the solution to the waste container

#### 13. Assessment of Significant Risks

- 1. Prevent the spillage when heating up the material
- 2. Fine powder may irritated respiratory system, so mask should be wore
- 3. Corrosive solvent should be place in a plastic container before transfer from one area to the other area
- 4. Dissolve of polymer/Li should be in the fume cupboard
- 5. Chemicals used and chemical wastes may lead to potential hazards
- 6. Corrosive materials e.g. sulfuric acid, may cause damage to the skin and cause burn.
- 7. Irritant materials
- 8. Li salts and polymers used in this project are corrosive and harmful.
- 9. Hot plate will be operated at high temperature.

#### 14. Safety Precautions

#### **Safety Training Required**

Safety training courses, included Chemical Safety and Hazardous, Waste Management training organized by the HSEO should be attended.

#### **Equipment Training Required**

Training with technician to use the glovebox and other machines should be arranged.

#### **Personal Protective Equipment**

Laboratory coat, safety glasses, nitrile gloves (TNTTM Blue, Ansell), brass tongs, heat gloves (while handling hot ash samples and reflux), cryogenic gloves (Cryoglove TM, Tempshield), face shield (while handling hot ash samples and liquid nitrogen) and dust mask (in the event of handling fine carbon) are worn throughout experiments.

Fume cupboard will be used when handling hazardous chemicals listed in the Chemical and Materials section.

In the event of emergency (e.g. chemical spillage) workers' actions will follow the standard procedures suggested by University's Chemical Safety training as well as Emergency Procedures.

#### Safety precautions of flammable liquids

1. Keep the flammable chemicals away from heat and ignition sources.

2. Do not leave the containers opened as vapors can flow along surfaces and travel a considerable distance to a source of ignition and flash back.

#### Safety precaution of handling powders

- 1. Face mask should be wear when handle the fine particles to prevent breath into the lung.
- 2. Be careful when handle the very fine powder, since might cause explosion

#### Warning sign required

- 1. All the chemicals will be stored based on the hazard categories with clear labeling.
- 2. The composition of the chemicals, date of receipt, use and expiration must be noted on label.
- 3. Chemical inventory will be minimized to reduce the overall risks.
- 4. Labels of high voltage and high current must be noted and be seen clearly.

#### **Emergency Procedures**

- 1. Ventilated area and wash spill site after material pickup is complete. Evacuate area.
- 2. If inhaled the toxic fumes, remove to fresh air. If breathing is difficult, give oxygen.
- 3. In case of skin contact, flush with copious amounts of water for at least 15 minutes. Assure adequate flushing by separating the eyelids with fingers. Call a physician.
- 4. If the situation is out of control, evaluate from the scene immediately. Alert others people to evaluate from the scene immediately. Inform SEPO and security unit promptly about the incident.

#### 15. Action in Case of Abnormal or Emergency Situation

In the event of emergency workers' actions will follow the standard procedures suggested by University's Chemical Safety training as well as Emergency Procedures. All abnormality during experiment shall be reported to laboratory technical staff immediately.

#### In case of Chemical Spill

If hazardous chemical spill in research laboratory, alert co-workers first and report to laboratory technical staff immediately. If safe to do so, confine the spill with appropriate materials. Turn off remotely all heat/ignition sources if flammable vapor is involved. Ask for assistance if necessary. Press the Emergency Ventilation bottom (do

not activate this bottom in case of fire). Inform the Security Control Centre by dialing 8999 from a safe location. Evacuate everyone in the affected area. Leave contaminated clothing and close the door. Activate local warning system to prevent others from entering the room. If possible, maintain a safe distance from the scene, keep the entrance or access routes in sight and help to prevent entry to the affected room. If conditions allow, remain to assist the emergency response team.

#### In case of Fire

When you hear fire alarm, remain calm and check if there is any sign of fire in the vicinity. If you see fire or smoke, or hear the announcement asking you to evacuate, follow the evacuation procedures. If there is no sign of a fire, stay alert and pay attention to announcement until the fire alarm is silenced. Evacuate if the alarm has sounded for more than two minutes.

If you discover a fire, activate the fire alarm by pressing the break glass fire alarm button. Report to Security Control Centre by dialing 8999. Alert other people. If Safe to do so, try to put out the fire by firefighting equipment. Do not take any personal risk. If the fire gets beyond your control, evacuate immediately by following the procedures below:

Close the door of the room on fire.

In case of someone is injured or ill:

Call Security Control Centre (SCC) by dialing 8999.

Call for Community Emergency Service directly by dialing (9)999 if the situation is urgent or serious and inform SCC subsequently.

Do not conduct rescue operation unless you know for sure how to perform a proper rescue or you know the situation is safe. Careless rescue operation may endanger the rescuers when, for example, the victim is inside a room filled with toxic gas, or is still in contact with live electricity.

Do not move an injured person, especially when there are signs of spinal injury or bone fracture, unless it is absolutely necessary to do so for safety reason.

Keep the injured or ill person comfortable, warm, and lying down.

Give First aid treatment if necessary:

- 1. Acid and alkali burns--flush with running water; use emergency shower if necessary. Do not attempt to neutralize.
- 2. Heat or cold burns--flush with cold water.

- 3. Chemical in eyes--flush eyes with emergency eyewash.
- 4. Major bleeding--apply direct pressure to the wound using a clean cloth.
- 5. Toxic gas inhalation--expose to fresh air.
- 6. Hydrofluoric acid exposure--use antidote immediately
- 7. Cyanide exposure--use antidote immediately

#### 16. CBME Risk Assessment Audit Declaration

See the attachment