

# WORK PERMIT

Department of Chemical and Biomolecular Engineering

化學工程及生物分子工程學系

Project Title: A New Class of Electrocatalysts for  
Hydrogen Evolution Reaction.

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# A New Class of Electrocatalysts for Hydrogen Evolution Reaction

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## 1. General Information

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Name of Researcher:	Nam Jae WOO Chae Young You Yuebin Liu
Name of Project Supervisors:	Dr. Minhua SHAO
Project Title:	A New Class of Electrocatalysts for Hydrogen Evolution Reaction
Research Area:	Chemical Engineering
Location:	Lab 7102
Proposed Start Date:	April. 2017

## 2. Experiment/Project Description

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### Objective:

The aims and the objectives of this project are:

- a.) to develop Me-PAN nanofibers (Me=Co, Fe, Ru, etc.) as catalysts for hydrogen evolution reaction in water splitting;
- b.) to optimize the preparation conditions to tune and control the structures of the catalysts;
- c.) to study the activity of the developed catalysts;
- d.) to improve the efficiency of hydrogen evolution reaction with use of the developed catalysts .

### Experiment Description:

To synthesis Me-PAN nanofibers and characterize the samples by XRD, TEM, SEM, etc.

To further evaluate their catalytic activities in a three-electrode cell.

### 3. Equipment List

Equipment	Location
Electronic balance	7102
Pipette (80µl, 1.5ml, 0.5ml)	7102
Stirring heater	7102
50 ml, 100 ml, 200 ml Volumetric Flask	7102
Beakers (of various volumes), test-tubes, sample holder bottles	7102
pH meter	7102
Beakers (of various volumes), test-tubes, sample holder bottles	7102
X-ray diffraction (XRD) equipment	2150
Transmission Electron Microscope (TEM)	2213, 2218
Scanning Electron Microscopy (SEM)	1125
Induced Coupled Plasma (ICP)	7101
Raman spectroscopy	7106&2153
Oven	7102 SC
Ultrasonic instrument	7102
Centrifuge	7101&6114 SC
Fume hood	7102
Potentiostat	7102
Furnace	7102
Home-made electrochemical cell	7102
Electrospinning unit	G031C

## 4. Experimental Procedures

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### 4.1 Synthesis of Me-PAN nanofibers

(4.1.1) Metal/PAN-based fibers were prepared by electrospinning a composite solution of both a desirable amount of metal precursors like (1) ruthenium(III) acetylacetonate, (2) iron(III) acetylacetonate, (3) cobalt(III) acetylacetonate and 0.5g (4) polyacrylonitrile (PAN) polymer solution dissolved in 14ml (5) dimethylformamide (DMF). The amount of ruthenium(III) acetylacetonate used was 10wt %. A composite solution was placed in a 20 mL syringe with a positively charged capillary tip with a diameter of 0.5 mm. The electrospun nanofiber composite web was collected by attaching it to aluminum foil wrapped on a metal drum rotating at approximately 300 rpm. The applied voltage was 15 kV, the distance between the syringe needle tip and collector was 18 cm, and the flow rate of the spinning solution was 1 mL / h .

(4.1.2) The electrospun fibers were stabilized by heating them to 220 ° C at a rate of 5 ° C / min and holding them at this temperature for 3 h in an air atmosphere. The stabilized fibers were carbonized by heating them to 800 ° C at a rate of 5 ° C / min and activated by supplying 30 vol % steam in a argon carrier gas for 1 h .

## 5. PROCEDURE TEMPLATE

Experimental Procedure No.	Experimental Procedure Description	Scale (Mass/Volume)	Location	Method
				New or Existing
4.1.1	0.5g of metal precursor and 0.5g PAN were dispersed in 14ml DMF. 15 kV voltage was added to obtain nanofibers.	Metal precursor:0.5g PAN:0.5g DMF:14ml	Fumehood -7102 Electrospinning unit-G031C	Existing
4.1.2	The electrospun fibers were stabilized at 220 ° C and carbonized at 800 ° C.		Tube furnace -7102	Existing

6. HAZOP Template							
4.1.1							
NO	HAZARD	HAZARD EFFECT	SEVERITY	PROBABILITY	RISK	MINIMISE RISK BY	RESIDUAL RISK
1	Contact with chemicals	Causes severe skin and eye burns.	H	M	H	Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood	L
2	Volatile chemicals inhalation	Harmful by inhalation.	H	M	H	Take the measurement within the fume hood	L
FINAL ASSESSMENT:						OVERALL RISK:	L

Remark: Severity – Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)  
 Probability – Unlikely:1; Possible:2; Very Likely: 3.

HAZOP Template							
Hazard and Operability Analysis							
4.1.2							
NO	HAZARD	HAZARD EFFECT	SEVERITY	PROBABILITY	RISK	MINIMISE RISK BY	RESIDUAL RISK
1	Contact with chemicals	Causes severe skin and eye burns.	H	M	H	Wear protective gloves, face shield and lab coats; Conduct experiments in the fume hood	L
2	Volatile chemicals inhalation	Harmful by inhalation.	H	M	H	Take the measurement within the fume hood	L
3	High temperature	Skin burn if touched	M	L	M	Set a warning sign and operate with protective gloves. Remove samples	L

4	Temperature run off	Fire risk	L	L	L	only when they have cooled to 60C.	L
FINAL ASSESSMENT:							OVERALL RISK:
Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality) Probability - Unlikely:1; Possible:2; Very Likely: 3.							L

Remark: Severity - Low:1 (Minor injuries, first aid); Medium:2 (Hospitalization, medical leave); High:3 (Serious injuries, fatality)  
Probability - Unlikely:1; Possible:2; Very Likely: 3.



## 7. Operating Conditions

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### Synthesis of Me-PAN nanofibers

- Pressure : Ar atmospheric
- Temperature : 20°C~800°C
- Flow rates : batch operations therefore not applicable

## 8. Services List

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Electricity (AC 220V, 50Hz)

Electrospinning unit

Double de-ionized water

Tap water

Compressed Argon

## 9. Chemicals List

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Number	Chemical	Purity	Quantity per Experiment
(1)	ruthenium(III) acetylacetonate	97%	0.5g
(2)	iron(III) acetylacetonate	97%	0.5g
(3)	cobalt(III) acetylacetonate	98%	0.5g
(4)	polyacrylonitrile (PAN)	--	0.5g
(5)	dimethylformamide (DMF)	99.9%	14ml

## 10. Biological Agents List

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N/A

## 11. Summary of Relevant Hazards and Incompatibilities

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Material	Summary of Hazards	Incompatibilities
ruthenium(III) acetylacetonate	Not a hazardous substance	Strong oxidizing agents
iron(III) acetylacetonate	Toxic and irritant	Strong oxidizing agents, Strong bases
cobalt(III) acetylacetonate	Danger	Oxidizing agents
polyacrylonitrile	Not a hazardous substance	Strong oxidizing agents
dimethylformamide	Irritant and harmful	Strong oxidizing agents

## 12. Waste List

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(1) The metal ions contained solution will be collected in the metal ions waste container (located at 7/F service corridor).

## 13. Assessment of Significant Risks

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### Synthesis of Me-PAN nanofibers

- Heat treatment of samples under high temperature

Protective gears such as lab coats and gloves are worn all the time when handling toxic and corrosive chemicals such as acids and Hydrazine monohydrate, care is to be taken when handling and transporting them and preparation from stock solution is done in fume hood to protect others. Flammables will be stored in flammables cabinet and away from heating source. Warning signs will be put in place when high temperature reaction is carried to warn other lab users.

## 14. Safety Precautions

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### *Safety Training Required*

- ☐ The researcher must attend regular courses (and in some courses obtain a passing result) offered by the Safety and Environmental Office of the University (HKUST) and understand thoroughly the safety concepts of these courses and be able to apply them before being allowed to conduct any experiments or use any of the equipment associated.
- ☐ The researcher must understand and be aware of all the procedures for handling, and in some cases dealing, with all possible emergency situations and scenarios.
- ☐ The researcher must undergo training in handling equipment and conducting experiments competently before performing it on his/her own.
- ☐ Equipment training offered by technical staff for nitrogen adsorption (Omnisorp 100CX), ICP (Perkin Elmer Optima), Elemental Analyser (Vario ELII Eliminator), TGA (Shimadzu TGA-50) are required before conducting the tests.
- ☐ XRD, XRF, XPS, TEM, and FTIR will be conducted by the technical staff of the MCPF.

### *General Laboratory Safety Rules*

It is HKUST policy to ensure that employees and students who are engaged in potentially hazardous operations receive both general and job-specific safety training prior to conducting these operations. Each department will establish a management scheme to implement this policy. Each supervisor will evaluate the nature of the work and will determine what safety training is needed for employees and students under his/her supervision.

General safety courses listed below cover a wide variety of safety issues. In addition to assigning employees and students to attend these courses, supervisors shall also provide job-specific safety training to ensure the safe performance of potentially hazardous operations.

Mandatory Courses (Chemical Safety II / Hazardous Waste Management, MC03; Pressure Safety, MC05; Chemical Safety I / Chemical Safety for Laboratory Users, MC07) and Discretionary Courses (Fire Safety and Fire-fighting Equipment, DC03) should be taken in terms of this project.

### *Personal Protective Equipment*

- ☐ Laboratory coat, safety goggles, and nitrile/polyvinyl alcohol gloves because of the presence of solvents such as toluene (from Instant Glove + CPC Database) as the outer layer with rubber gloves as the inner layer must be worn at all times when performing the experiments, transporting, handling and

cleaning chemicals and equipment and clearing chemical spills.

☐ Dust mask should be worn when collecting and weighing solid particles.

☐ Heat resistant gloves will be worn when handling hot items..

#### *Handling Organic Compounds*

Personal Protective Equipment should be used when handling organic compounds. When disposing of organic waste, organic solvents with halogens (e.g. trichloromethane, trichloroethylene, and dichloromethane) should be collected in "Halogenated Solvents" container and other liquid organic compounds with halogens. "Non-halogenated Solvents" container is for organic solvents and other organic compounds without halogens (e.g. acetone, hexane, and petroleum ethers).

#### *Handling Flammable Liquid*

Carefully read the manufacturer's label on the container of any flammable liquid before storing or using it. Practice good housekeeping in flammable liquid storage areas. Clean up spills immediately then place the cleanup rags in a closed, bottom ventilated, metal container. Only use approved metal safety containers or the original manufacturer's container to store flammable liquids. Keep the containers closed when not in use; stored away from exits or passageways.

#### *Handling Heat Process*

Be aware of heat process and mind high temperature equipment, especially the temperature controller. Don't touch the high temperature unit directly.

## **15. Action in Case of Abnormal or Emergency Situations**

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### **(A) In case of loss of containment or accidental spillage**

#### **(1) Minor spillage (<100mL)**

Alert co-workers

If safe to do so, confine the spill with appropriate material and/or turn off remotely all heat/ignition sources if flammable vapour is involved.

Ask for assistance is necessary.

Press the emergency ventilation button (do not activate this button in case of fire).

Inform the security office at x8999 or 23588999 with mobile phone when it is safe to do so.

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.

#### **(2) Solid chemicals**

Alert co-workers

If safe to do so, confine the spill with appropriate material and/or turn off remotely all heat/ignition sources if flammable vapour is involved.

Ask for assistance is necessary.

If confident, clean the spillage properly, being protected by Personal Protective Equipment.

#### **(3) Major spillage (>1L)**

Alert co-workers

If safe to do so, confine the spill with appropriate material and/or turn off remotely all heat/ignition sources if flammable vapour is involved.

Ask for assistance is necessary.

Press the emergency ventilation button (do not activate this button in case of fire).

Inform the security office at x8999 or 23588999 with mobile phone when it is safe to do so.  
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.

## **(B) Fire-fighting measures**

### **When the fire alarm is heard**

- Check if there is any sign of fire in the vicinity.
- If there is fire or smoke, or there is an announcement to evacuate, then evacuate to the assembly point as far as practicable and report to the fire & safety officer.
- 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 the buzzer sound which indicates fire alarm is activated in an adjacent fire zone is heard, stay alert and pay attention to announcement.
- If both the buzzer and the fire alarm are heard, treat as if the fire alarm is heard.

### **If a fire is discovered**

- Perform emergency shut down procedures if possible.
- Activate the fire alarm by pressing the breakglass fire alarm button.
- Report to Security Control Centre by dialling 8999.
- Alert other people. If safe to do so, try to put out the fire by fire fighting equipment.
- Do not take any personal risk. If the fire gets beyond control, evacuate immediately as listed above.
- Close the door of the room on fire.

### **Fire fighting equipment**

- Water from the hose reels is good for wood and paper fire, structural fire, but not for oil, electrical or metal fire.
- The most common fire extinguishers on campus is the carbon dioxide type (black container) which are good for general purposes, including oil and electrical fire.
- Some laboratories have dry powder fire extinguishers (blue container), which are good for chemical fire, including metal fire.
- Sand can be used to contain flammable liquid as well as put out a fire, including metal fire.
- Fire blanket can be used when someone's clothing catches fire.

### **Evacuation procedures**

- Remain calm. Walk; do not run, especially when travelling on staircases.
- Immediately leave the building and go to the assembly point using the nearest exit.
- Try to help those who may have difficulties traveling such as disabled and pregnant persons.
- Do not use the lifts.
- Report to your Fire & Safety Officer at the assembly point as far as practicable.
- Do not return to the building until permission is given by the Fire Services Department Officer in charge of the scene.

**When clothing is on fire**

- Do not run
- Drop to the floor and roll to extinguish the fire.
- If fire blanket is available, wrap around body to help smother the fire.