AN INVESTIGATION OF CYLINDRICAL LITHIUM-ION BATTERY THERMAL RUNAWAY PREVENTION USING LIQUID-SUBMERGED TECHNIQUE

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A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENT FOR THE DEGREE OF MASTER OF ENGINEERING
IN AUTOMOTIVE AND ADVANCED TRANSPORTATION ENGINEERING
SCHOOL OF ENGINEERING
KING MONGKUT'S INSTITUTE OF TECHNOLOGY LADKRABANG
YEAR 2023

KMITL-~~2023-EN-M-037-029~~

THESIS TITLE An Investigation of Cylindrical Lithium-ion Battery Thermal Runaway Prevention Using Liquid-submerged Technique
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ABSTRACT

This study investigates the effects of submerging fully charged lithium-ion battery (LIB) cells and modules in various liquids, including natural seawater (NSW), synthetic seawater (SSW), single salt solutions, deionized water, and Thai tap water, in order to better understand thermal runaway prevention and fire suppression in LIBs. The experiments involved submerging LIBs in the liquids for 3 hours or until the LIB terminal was destroyed, at which point the voltage reduction was manually measured. The terminals were then removed from the liquids and examined for corrosion using a camera. In addition, the filtered solutions were analyzed using X-ray diffraction and Fourier transform infrared spectroscopy to evaluate the possible reactions and organic electrolyte leakage. Finally, a single cell was subjected to an overheating abuse test to trigger thermal runaway, and the liquid submersion technique was used to suppress the resulting fire/explosion. The results demonstrated a slight discharge rate in deionized water and Thai tap water, with minimal terminal corrosion. However, NSW and SSW resulted in rapid discharge and significant terminal corrosion on the positive terminal. The liquid submersion technique was effective at dropping the voltage to 0 V, which is considered safe for stopping hazardous consequences from thermal runaway. This research also has implications for the development of an upscaled fire suppression system for high voltage battery packs.

Keywords: Lithium-ion battery safety, Thermal runaway prevention, Submersion fire suppression, Battery submersion, Seawater submersion

ACKMOWLEDGEMENT

I could not have undertaken this journey without the scholarship from Thailand Advanced Institute of Science and Technology and Tokyo Institute of Technology program (TAIST-Tokyo Tech). My thesis would not have been completed without this financial support from them.

I would like to express my deepest gratitude to my advisor Asst. Prof. Dr. Chinda Charoenphonphanich from King Mongkut’s Institute of Technology Ladkrabang (KMITL), Dr.-Ing Manop Masomtob and Dr. Nattanai Kunanusont form National Science and Technology Development Agency (NSTDA) and Prof. Dr. Shuichiro Hirai from Tokyo Institute of Technology, Japan for their intangible support, patience, and kindly guidance during this work.

I am also thankful to the Energy Storage Technology Research Team (ESTT), research staff of National Energy Technology Center (ENTEC), and all of lecturers for intangible support which includes the suggestions, the device, facilities, and research fund.

Lastly, I would like to mention my family, especially my parents, my partner and my friends for their love, emotional support and always be there for encouragement. Their belief in me has kept my spirits and motivation high during this process. Moreover, I would also like to thank all of MP144 members/staffs for all entertainment and practical support through this research.

Pongkorn Meelapchotipong

TABLE OF CONTENTS

LIST OF TABLES

Table Page

LIST OF FIGURES

LIST OF DEFINITIONS

ACM	Polyacrylate
BTMS	Battery thermal management system
CC-CV	Constant current constant voltage
CMC	Carboxymethyl cellulose
DEC	Diethyl Carbonate
DMC	Dimethyl carbonate
EC	Ethylene carbonate
EV	Electric vehicle
FTIR	Fourier transform infrared
GBL	gamma-butyrolactone
HMPN	Hexamethyl cyclotriphosphazene
ISC	Internal Short Circuit
LCO	Lithium Cobalt Oxide(LiCoO2)
LFP	Lithium Iron Phosphate(LiFePO4)
LIB	Lithium-ion battery
LMO	Lithium Manganese Oxide (LiMn2O4)
LTO	Lithium Titanate (Li2TiO3)
NCA	Lithium Nickel Cobalt Aluminum Oxide (LiNiCoAlO2)
NMC	Lithium Nickel Manganese Cobalt Oxide (LiNiMnCoO2)
PAN	Polyacrylonitrile
PC	Propylene carbonate
PEO	Polyethylene Oxides
PMMA	Polymethyl methacrylate
PTFE	Polytetrafluoroethylene
PVDF	Polyvinylidene fluoride
RTIL	Room temperature ionic liquid

SBR	Styrene-Butadiene Rubber
SoC	Stage of charge
TFP	Tris (2,2,2-trifluoroethyl) phosphite
TMP	Trimethyl phosphate
TR	Thermal runaway
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 RESEARCH BACKGROUND

The Lithium-ion battery (LIB) is commonly used as the main power source of Electric Vehicles (EVs) in recent years due to its high specific energy, light weight long life cycles, low self-discharge rate, and low memory effect comparing to lead-acid battery and Nickel-based battery [1]–[3]. However, the internal temperature of LIB becomes higher when operated on high charge/discharge rate, leading to thermal runaway (TR) which is a rapid and unstoppable increase of temperature in a sort of chain reaction and battery fire explosion [4], [5].

There have been many Electric Vehicle (EVs) in recent years including electric boat accidents resulting in fire and explosion in recent years. In October 2019, the hybrid ferry "Ytterøyningen" caught fire and exploded 12 hours after fire extinguishing in Norway. Fires occurred when the ferry only ran on diesel engine then spread to battery compartment leading to battery thermal abuse condition [6], [7]. Furthermore, there was a fire that occurred near the battery compartment of a hybrid-electric boat in March 2021. After fire extinguishing, firefighters battled smoke and battery elevated temperature for more than 24 hours to prevent fire re-ignition and explosion [8].

In the case of electric ferry operated in the ocean, using seawater, which is electrolyte solution, as fire extinguishant seems like a simple solution to ensure that LIB is burned out. For this reason, submersion battery into liquid and electrolyte solution is an effective method to suppress LIB fire. It offers not only improved thermal performance but also hinder the exothermic reaction and prevent reignition [9]–[13]. However, the liquid waste problem is still the main concern and need further research information to optimize conditions.

This research focused on submerging single cell to study possibility of LIB fire suppression using synthetic seawater, DI water and Thai tap water to study the LIB fire after liquid submersion, discharge rate, cell deconstruction, environmental pollution after process and effectiveness of fire suppression using liquid submersion. In this study, the corrosion and cell deconstruction which lead to organic electrolyte leakage are analyzed. Then, safety submersion time, which is the maximum time length to submerge LIB before serious corrosion occurs is provided to enlighten the knowledge on fire suppression from LIB using liquid submersion technique. Moreover, single cell was subjected to overheat abuse condition from external heat source before using liquid submersion technique to prevent Thermal runaway and suppress fire/explosion from Lithium-ion battery.

1.2 GOALS AND OBJECTIVES

- To represent a series of experiments to observe fire/explosion from cylindrical Lithium-ion battery liquids submersion, thermal runaway from overheated abuse condition, and preventing/suppression effect from liquid-submerged technique.
- To present the consequences of cylindrical Lithium-ion battery liquids submersion with/without thermal abuse condition.
- To investigate the possibility of cylindrical Lithium-ion battery thermal runaway prevention/fire suppression using full coverage liquid submersion suppression technique for safety reasons of Lithium-ion energy storage system.

1.3 SCOPE OF THE RESEARCH

- The TORIYAMA N18650CL-29 commercial 18650 Lithium-ion cells with NMC ($LiNiMnCoO_2$) as a cathode of the cell is used in these experiments.
- The effect of state of health (SOH) is not considered in this research.
- Only single cell submersion is considered in these experiments.
- Synthetic seawater (SSW) from ASTM D1141-98 standard can be used as Natural seawater (NSW) collected from Bangsaen Beach in Chonburi province, Thailand.
- Thai tap water used in this experiment was collected from Pathum Thani province in Thailand.
- Heating pad temperature is controlled by MAXTHERMO MC-5638 temperature PID controller with type-K thermocouple.
- Suppression system was triggered by digital voltage controller with relay output control loads.

1.4 EXPECTED BENEFITS

- Confirming that liquid submersion technique can be used to prevent Thermal runaway and suppress fire/explosion from Lithium-ion battery.
- Providing information for choosing types of liquids to submerge Lithium-ion cell/module that applied to electric vehicle.
- To scale up into Lithium-ion battery module TR/fire suppression using liquids submersion system

1.5 THESIS LAYOUT

This research is divided as follows.

CHAPTER 1 discusses research background, scope of this research, and the expected benefits.

CHAPTER 2 presents literature reviews related to this research. This chapter addresses the principle of Lithium-ion battery, battery abuse condition and Thermal runaway, electric vehicle accidents, Lithium-ion battery fire suppression, and Lithium-ion battery liquid submersion.

CHAPTER 3 presents the experimental methodology. A series of experiments, including liquids submersion and fire suppression, are presented.

CHAPTER 4 presents an approach to submerge a single Lithium-ion battery cell into liquids such as synthetic seawater, DI water and Thai tap water to observe behavior of the cell submerged in liquids. The voltage of cell and the pH of the solution are discussed. Moreover, the solution characterizations are analyzed using XRD and FTIR.

CHAPTER 5 presents the results of Lithium-ion battery thermal runaway prevention and fire suppression using liquid-submerged technique. The cell was overheating making Lithium-ion battery subjected to thermal abuse condition and thermal runaway. The voltage and surface temperature of the cell are observed throughout the experiments to obtain voltage behavior and critical temperature. Then, the results of liquid submersion experiment are discussed.

CHAPTER 6 summarizes the results and main achievement of this research and suggests future work and applications for applying this research knowledge.

CHAPTER 2

LITERATURE REVIEWS

2.1 LITHIUM-ION BATTERY

Lithium-ion battery (LIB) is the electrochemical cell that connects in parallel and/or series to perform the required capacity and voltage respectively. It stores electrical energy in electrodes that made of lithium-intercalation compound with chemical reactions occurred between layered positive electrode and graphite negative electrode separated by separator and electrolyte solution containing dissociated salts. During charging process, the lithium-ions are deintercalated from the layered cathode host, which is positive electrode, then intercalated between the graphite layer in anode. On the other hand, the discharging process is a reverse of charging process and electron pass around the external circuit to power the external systems as shown in Figure 2.1 [14].

There are various types of LIB configurations including cylindrical, prismatic and pouch cell as shown in Figure 2.2. Cylindrical cells are the most products that follow standard model in term of the size of the cell. The standard size for metal-encased cylindrical cells is 18650, which is 18 millimeters in diameter and 65 millimeters in tall, 21700, 26650, 32700 and 4680. Prismatic cells have various cell capacities used in cell phones, laptops, and electric vehicles. It can improve space utilization and has flexible pack design but expensive in manufacturing. The pouch cell offers simple, flexible, and lightweight pack design. It has the highest efficiency in terms of space utilization. On the other hand, according to the pouch cell has less metal casing, it needs a space for additional support and swelling [15].

Figure 2.3 shows the typical structure of 18650 cylindrical cell and its components. The negative terminal, which is a battery can, is connected to graphite layer anode of the electrochemical system. On the other hand, the positive terminal is connected to layered positive electrode with plastic insert separate positive terminal and negative terminal. Usually, the positive terminal contains the top cap, which is mainly made of steel, and a vent disk with groove and bottom disk layer. During normal operation and TR condition, a pre-made through-holes allows gas and fluids flow at the bottom disk layer. Then, the vent disk blocks gas and fluids generated from operation and failure until pressure difference applied to vent disk breaks the pre-made slot at vent disk allowing gas and fluids to escape called venting mechanism. It can reduce the internal pressure, decelerate the TR process, and prevent the cells from rupture and explosion [16].

Table 2.1 summarizes the typical components of lithium-ion batteries which is including cathode materials, anode materials, electrolytes, binders, and separators. Lithium Iron Phosphate (LFP) is the safest cathode material due to its thermal stability and non-toxicity but in terms of energy density, LFP is lower than the other materials. In anode material, carbon and graphite are commonly used because of its ability to reversely accommodate a significant amount of lithium. The electrolyte in LIB is mainly based on organic solvents which are often highly flammable. Moreover, it's commonly mixed with various carbonates and dissolved salt such as $LiPF_6$. For the binder of lithium-ion batteries, polyvinylidene fluoride (PVDF) is widely used. Additionally, thin microporous polyolefin membrane that made of polyethylene (PE), polypropylene (PP), and laminated PP/PE/PP are mostly used [17].

In electric vehicles powered by LIB, multiple cells are connected in parallel and/or series to form battery module. Then, battery modules are connected in parallel and/or series to meet the required voltage and capacity which is called a battery pack [18] as shown in Figure 2.4.

Table 2.1 List of typical components in Lithium-ion battery [17]

Cathode	LMO, LCO, NCA, NMC, LFP, ECPs
Anode	Graphitic carbons, Hard carbons, Synthetic graphite, LTO, Tin-based alloys, Silicon-based alloys
Electrolyte salts	$LiPF_6$, $LiClO_4$, $LiAsF_6$, $LiCF_3SO_3$, $LiBF_4$
Electrolyte solvents	DMC, EC, DEC, PC, gamma-GBL, RTIL's
Flame retardants	HMPN, TMP, TFP
Gel precursor	PEO, PAN, PVDF, PMMA, PTFE
Binder	PVDF, SBR, Glass Fiber, CMC, ACM
Separator	Polypropylene, Polyethylene, Cellulosic paper, Nonwoven fabrics, Ceramic

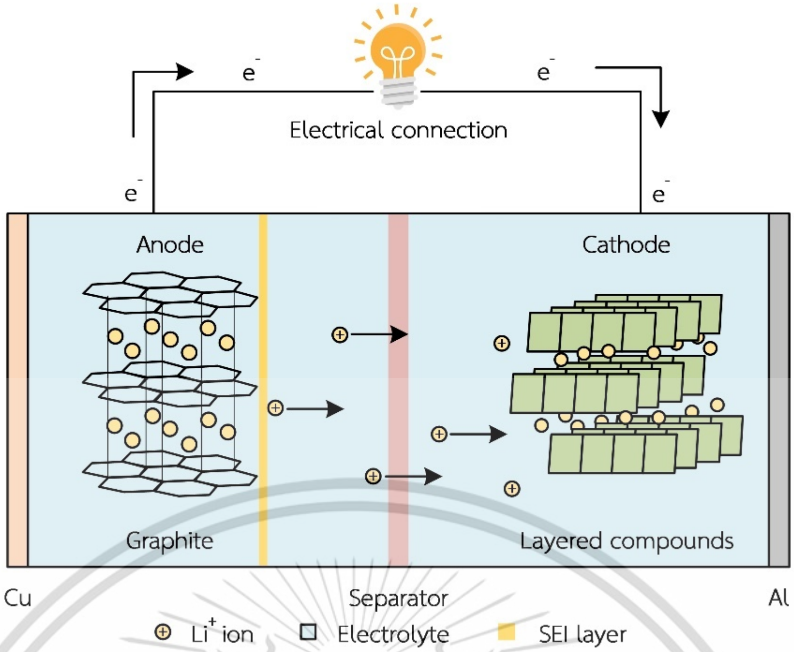


Figure 2.1 Schematic of a traditional Lithium-ion battery cell during discharge adapted from [14]

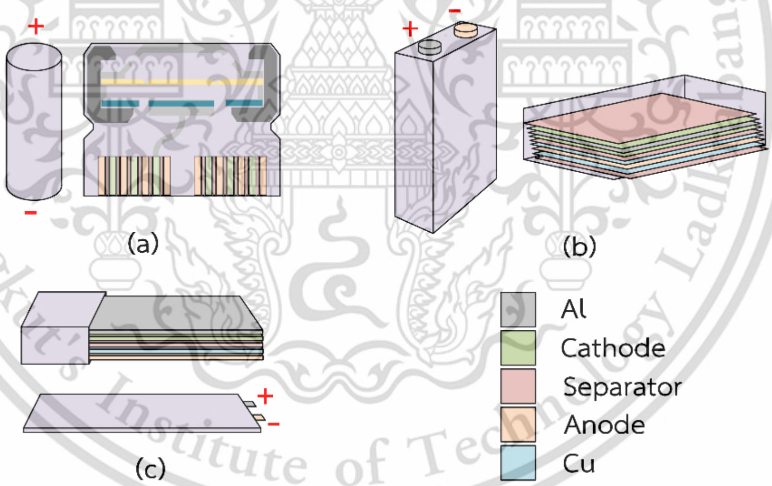


Figure 2.2 Lithium-ion battery cell configurations (a) cylindrical cell (b) prismatic cell and (c) pouch cell adapted from [15]

2.2 BATTERY ABUSE CONDITION AND THERMAL RUNAWAY

2.2.1 Lithium-ion battery abuse condition

The abuse condition can be categorized into three categories including mechanical abuse, electrical abuse, and thermal abuse as shown in Figure 2.6. However, all the abuse conditions mentioned above lead to battery internal short circuit (ISC) which is most common among various conditions. The ISC occurs when positive electrode and negative electrode of the cell contact with each other resulting in massive heat generation from the electrochemical energy stored inside the cell. It can happen from the defects of manufacturing and abuse conditions of each cell. The types of ISC can be categorized into two categories based on the root causes which are conductor penetrating the separator and the shrinkage or cracks of separator [2], [18].

The conductor penetrating, called mechanical abuse, comes from penetration into battery pack after crush resulting in dangerous consequences such as the leakage of organic electrolyte inside the cell causes battery fire and explosion [3], [18].

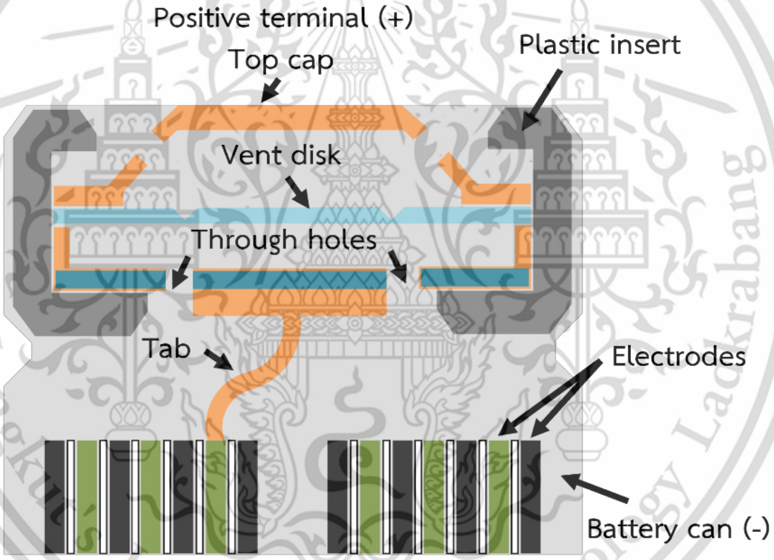


Figure 2.3 Typical structure of cylindrical Lithium-ion battery adapted from [16]

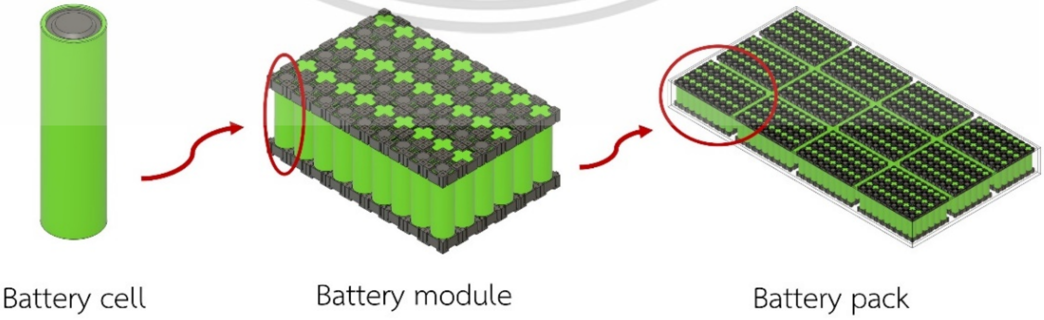


Figure 2.4 Schematic of cylindrical cell, battery module and battery pack

The shrinkage or cracks of cell's separator comes from the electrical abuse and thermal abuse condition. The electrical abuse is caused by external short circuit, overcharge, and overdischarge condition. The external short circuit occurs when electrodes relate to the difference of voltage. However, the heat released from external short circuit is small and does not heat the cell, but it can be reduced by protective electronics devices such as fuses, positive thermal coefficient (PTC) devices, magnetic switches, and bimetallic thermostats. The cell's overcharge occurs when the cell's voltage exceeds the cell's maximum voltage due to the failure of battery management system (BMS) to monitor and stop battery charging process resulting in ISC from lithium dendrite developed inside the cell. It can be harsher than any other abuse condition because excessive energy is filled into the cell resulting in heat and gas generation. Also, overdischarge condition as shown in Figure 2.5 occurs when BMS fails to monitor the voltage of any cell resulting the lowest voltage cell will be overdischarge. During overdischarge, the voltage of overdischarge cell becomes negative resulting in abnormal heat generation, solid electrolyte interface (SEI) decomposition, and gas generation like CO and CO_2 caused the swell of the cell [2], [3], [18].

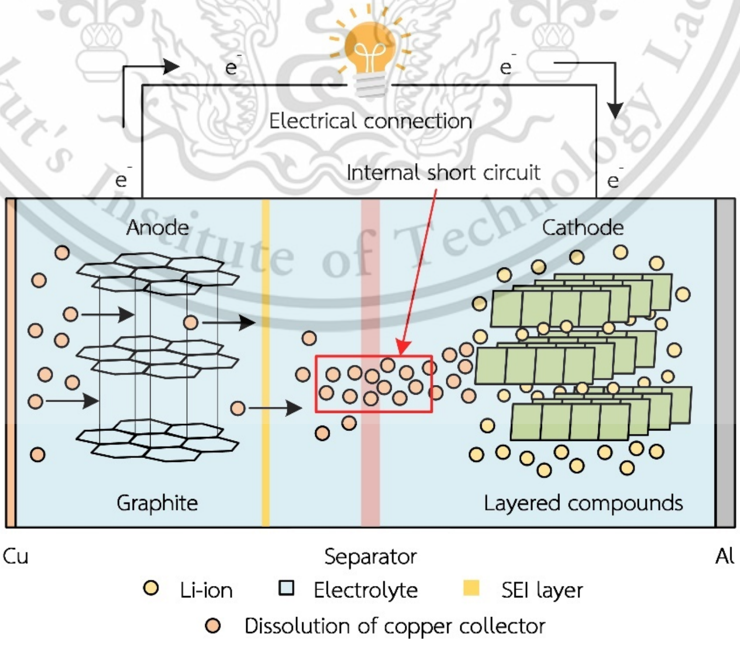


Figure 2.5 Overdischarge mechanism adapted from [3]

Thermal abuse is a direct cause of TR due to self-overheating and the heat from external heating sources. Self-overheating can occur by the contact loose of the connector generating local heat at area because of high current passing. When lithium-ion batteries are subjected to high temperatures, the separator collapses and melt resulting in the trigger more heat from exothermic reactions within positive and negative inside the cell [2], [3], [18].

2.2.2 Thermal runaway mechanism

Thermal runaway (TR) occurs when the temperature inside the cell rises continuously and reaches the critical temperature by heat generated from exothermic reactions inside the cell exceeds the heat dissipation rate [18]. This phenomenon can be triggered by various factors, such as heat generation during normal operation, reactions at the negative and positive electrodes, electrolyte and binder decomposition and combustion, as well as separator melting.

During normal operation, the reversible heat generation occurs from electrochemical reactions as shown in Equation (2.1 . On the other hand, the irreversible heat is generated from ohmic loss, charge-transfer and mass transport limitation inside the cell as shown in Equation (2.2). So, the total heat generation inside the cell is the summarize between reversible and irreversible heat generation as shown in Equation (2.3) where I is the current in Am^{-3}, T is the absolute temperature in K, a_s is specific interfacial area of the electrode in m^{-1}, J is the transfer current resulted from the intercalation or deintercalation of lithium in $mol\, m^{-2}s^{-1}$, and F is the Faraday constant in $C\ mol^{-1}$.

$$Q_{rev} = -IT\left(\frac{\partial E_{oc}}{\partial t}\right) = -a_s JFT\left(\frac{\partial E_{oc}}{\partial T}\right) \tag{2.1}$$

$$Q_{irr} = I(E_{oc} - E) = a_s JF(E_{oc} - E) \tag{2.2}$$

$$Q_{total} = Q_{rev} + Q_{irr} \tag{2.3}$$

The first stage of TR occurs from the breakdown and decomposition of SEI at internal temperature approximately 80-120 °C. The SEI is a thin layer formed on negative electrode material protecting the negative material of the cell from further reactions of the electrolyte as shown in Figure 2.1. It mainly contains two parts which are stable parts like LiF and Li_2CO_3, and metastable components such as $ROCO_2Li$, $(CH_2OCO_2Li)_2$, ROLi and oxygen-containing polymeric species. The decomposition of SEI can release CO_2 gas as shown in equation (2.4) [18] and can occur at temperature lower than 80 °C and might be started from 57 °C [3].

$$(CH_2OCO_2Li_2)_2 \rightarrow Li_2CO_3 + C_2H_4 + CO_2 + \frac{1}{2}O_2 \tag{2.4}$$

The decomposition of SEI layer not only produces heat to the cell, but also follows the reactions between the negative electrode and electrolyte. Anyway, after the decomposition of SEI layer occurred at higher temperatures, the SEI regeneration occurs from the product of the reactions between intercalated lithium and electrolyte from decomposed SEI. These reactions occur at approximately 120 – 250 °C. However, the SEI decomposition does not stop because of the SEI regeneration reactions while the SEI regeneration is not boost because of the negative electrode material surface still covered by SEI layer as shown in Figure 2.7. After that, the breakdown of SEI decomposition and SEI regeneration occur at approximately 250 °C of higher due to the collapse of carbon/graphite negative electrode material [3].

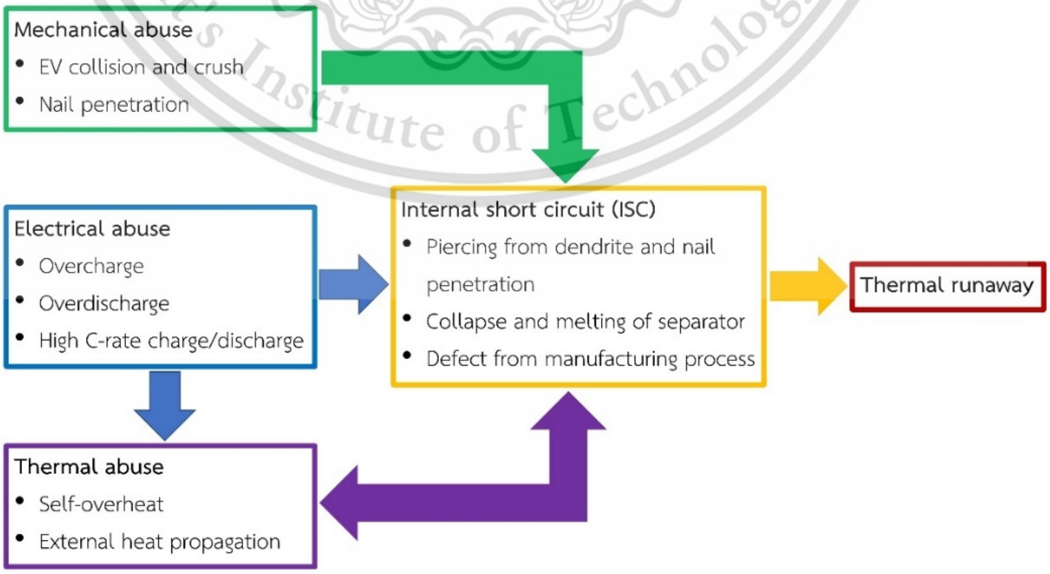


Figure 2.6 The common lithium-ion battery failure and abuse condition

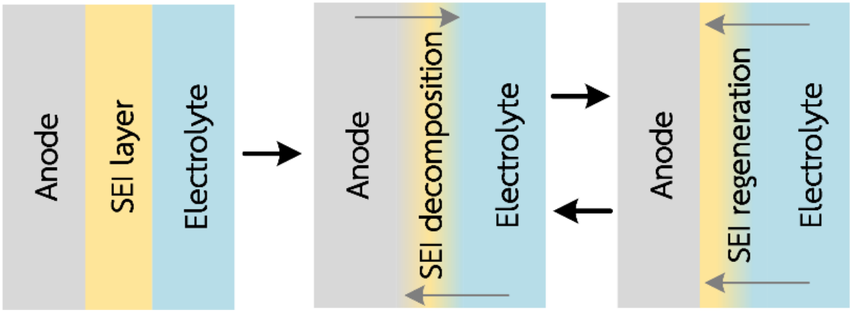


Figure 2.7 SEI decomposition and regeneration reaction adapted from [3]

The reactions from positive electrode of the cell can occur when the internal temperature exceeds onset temperature of the positive electrode material decomposition. Thermal stability depends on the type of positive electrode material in each cell. LFP is the most thermal stability in any of materials followed by LMO, NMC111, NCA and LCO respectively. In addition, the NMC properties depend on the ratio of its main composition which are Nickel, Manganese and Cobalt. The greater Nickel composed in positive electrode material, the more electric capacities obtained inside LIB, but the more Nickel presented in material, the lower thermal stability and onset temperature of cell will be. However, most of positive electrode materials decomposition generates oxygen gases as shown in equations (2.5) – (2.10) caused massive heat generation due to the reactions between the oxygen and flammable electrolyte. In this research, LIB with NMC as positive electrode material is used in the experiments.

$$Li_xCoO_2 \rightarrow xLiCoO_2 + \frac{1-x}{3}Co_3O_4 + \frac{1-x}{3}O_2 \tag{2.5}$$

$$\begin{aligned} Li_{0.36}Ni_{0.8}Co_{0.15}Al_{0.05}O_2 \rightarrow 0.18Li_2O + 0.8NiO + 0.05Co_3O_4 \\ +0.025Al_2O_3 + 0.372O_2 \end{aligned} \tag{2.6}$$

$$Li_{0.35}(NiCoMn)_{\frac{1}{3}}O_2 \rightarrow Li_{0.35}(NiCoMn)_{\frac{1}{3}}O_{2-y} + \frac{y}{2}O_2 \tag{2.7}$$

$$LI_{0.2}Mn_2O_4 \rightarrow 0.2LiMn_2O_4 + 0.8Mn_2O_4 \tag{2.8a}$$

$$3Mn_2O_4 \rightarrow 2Mn_3O_4 + 2O_2 \tag{2.8b}$$

$$LiMn_2O_4 \rightarrow LiMn_2O_{4-y} + \frac{y}{2}O_2 \tag{2.8c}$$

$$LiMn_2O_4 \rightarrow LiMnO_2 + \frac{1}{3}Mn_3O_4 + \frac{1}{3}O_2 \tag{2.8d}$$

$$Mn_2O_4 \rightarrow Mn_2O_3 + \frac{1}{2}O_2 \qquad (2.9)$$

$$2Li_0FePO_4 \rightarrow Fe_2P_2O_7 + \frac{1}{2}O_2 \qquad (2.10)$$

Furthermore, the electrolyte inside LIB can decompose and combust causing TR during operation since electrolyte inside the cell is mainly composed of flammable substances. $LiPF_6$ decomposition can generate HF as shown in equation (2.11). The oxidation reactions of organic electrolyte can release CO_2 and CO gases at temperature exceed 200 °C as shown in equation (2.12) and (2.13). The generated gases make internal pressure inside the cell rise resulting in triggering the opening of safety valve. During cell venting, the release gases may ignite increasing the hazard caused by TR.

$$LiPF_6 \leftrightarrow LiF + PF_5 \qquad (2.11)$$

$$2.5O_2 + C_3H_4O_3(EC) \rightarrow 3CO_2 + 2H_2O \qquad (2.12a)$$

$$6O_2 + C_5H_{10}O_3(DEC) \rightarrow 5CO_2 + 5H_2O \qquad (2.12b)$$

$$3O_2 + C_3H_6O_3(DMC) \rightarrow 3CO_2 + 3H_2O \qquad (2.12c)$$

$$4O_2 + C_4H_6O_3(PC) \rightarrow 4CO_2 + 3H_2O \qquad (2.12d)$$

$$O_2 + C_3H_4O_3(EC) \rightarrow 3CO + 2H_2O \qquad (2.13a)$$

$$3.5O_2 + C_5H_{10}O_3(DEC) \rightarrow 5CO + 5H_2O \qquad (2.13b)$$

$$1.5O_2 + C_3H_6O_3(DMC) \rightarrow 3CO + 3H_2O \qquad (2.13c)$$

$$2O_2 + C_4H_6O_3(PC) \rightarrow 4CO + 3H_2O \qquad (2.13c)$$

In addition, the binder can react with positive electrode material, negative electrode material and Li_xC_6 when temperature is above 260 °C. For example, the negative electrode intercalated with lithium-ions can react with PVDF.

The melting of separator inside the cell can cause ISC resulting in TR. The separator is commonly based on Polyethylene (PE) and Polypropylene (PP) materials which melting point is approximately 130 °C and 170 °C respectively. During the melting, holes in the separator are closed making the difficult of lithium-ion transfer inside the cell. It can display a collapse phenomenon by the increasing of cell resistance. To prevent the shutdown behavior with a little shrinkage of separator, the tri-layer PP/PE/PP was synthesized. So, the collapse of the PP/PE/PP separator is the same as PP separator. Moreover, Al_2O_3 and SiO_2 are the most coating ceramic making separator collapse temperature increase to around 200 °C to 260 °C.

Battery voltage drops rapidly due to the delamination of the battery electrodes. At the initial stage, the cell's voltage was almost unchanged at initial stage. However, a sharp voltage drop occurs only 15-40 seconds ahead of uncontrolled temperature. Anyway, the voltage drop phenomenon is not suitable as a warning signal to prevent TR. Due to the TR can be triggered by high temperature, it is necessary to track the battery temperature change to characterize the thermal behavior of the battery during operation and TR process [19].

2.3 ELECTRIC VEHICLE ACCIDENTS

There are some Electric Vehicle (EV) fire and accidents reported in recent years as shown in Table 2.2. Most of the accidents are from China, USA, and Norway. The normal causes are vehicle self-ignition while being parked and driven, fire during charging process, and fire after crash resulting in ISC [20]. The most possible cause of the EV accidents in Table 2.2 is from ISC followed by mechanical abuse from crash. Therefore, battery defects from manufacturing is also possible causes pushed EV ended up with fire and explosion. However, the cause of fire and explosion in some accidents could not be clearly identified because the accidents happened while the EV car was drove and parked without connecting to charging system. These EV accidents have raised LIB safety issues and LIB fire extinguish concerns.

2.4 LITHIUM-ION BATTERY FIRE SUPPRESSION

There are various kinds of fire extinguishants which can suppress LIB fire. However, it is not clear which types of fire extinguishants are the most suitable for LIB fire suppression. There are many types of extinguishant represented in [20]–[22] as shown in Table 2.4. Water-based extinguishants are the most cost-effective to suppress LIB fire. It successfully suppresses LIB fire, but gases and smokes still occur after LIB fire extinguished and battery re-ignition might occur after extinguished. Besides, water can react with $LiPF_6$ electrolyte forming harmful Hydrofluoric Acid (HF) and produce hydrogen gas which is combustible [20], [21]. However, extinguishing LIB fire by water needs large amount of water according to the report from captain fire-fighter in accident No. 5 stated in Table 2.3 that less than 300 gallons of water and foam was applied to suppress EV fire. Foam extinguishants suppress LIB fire faster than water by sealing surface and putting barrier between flammable vapor and hot surface/fuel. Nevertheless, the cooling effect is less effective than using water. Power and dry powder extinguishants are suitable for single cell extinguishing rather than battery module extinguishing because of the high LIB surface temperature. CO_2 extinguishants are not an ideal LIB fire extinguishant due to their low cooling capacity. It cannot cool the battery temperature down and re-ignition is often observed. Halon-based extinguishants do not offer cooling performance and may leave any residue. It could put out the LIB fire but re-ignite might occur. HFC-277ea and $C_6F_{12}O$ (Novec 1230 rapidly put out the LIB fire within 30 seconds but continuous spraying on LIB surface is necessary to prevent re-ignition. Lastly, aerosol extinguishant has low environmental impact, low toxicity, non-conductive and low residue. It rapidly extinguishes LIB fire in closed place, but re-ignition problem is still its biggest challenge.

There are many EV car accidents in recent years around the world, which ended up in LIB fire and explosion then used large amount of water to suppress the fire as shown in Table 2.3. The comprehensive comparison indicates that water-based extinguishants are the most suitable for LIB fire extinguishing [21]. There are many types of water-based extinguishants presented in recent years, including water jet, water spray or sprinkler, water with added surfactants, water mist (WM) and liquid submersion as shown in Table 2.5. The water jet method applies a stream of water directly to LIB fire. It provides cooling and inhibits battery re-ignition. Water spray or sprinkler is a spray of fine water droplet (above 1,000 μm). The spray droplets penetrate the fire plume and cool surface temperature down leading to reducing surface temperature effectively. Nevertheless, it does not prevent the propagation of TR and affects gas production during TR. Water with surfactants shows better performance by decreasing water's surface tension. WM method has less water consumption, low cost, no toxicity, low electrical conduction, and environmentally friendly due to its droplet size under

1,000 µm. The smaller droplet size leads to the greater absorption of heat energy due to the larger surface area to volume ratio. It has an effective TR prevention in multiple batteries. However, WM method still has a problem in the uniformity of water mist, which hardly reaches the surface of LIB and easily affected by ventilation condition [5], [21], [23]. Lastly, the liquid submersion as shown in Figure 2.8 provides the greatest temperature uniformity and safely maintains target temperature of the cell [9], [24]. It can prevent thermal propagation of the TR cell to adjacent cell and prevents hazardous from EV accidents because of the coolant can dissolve flammable gas resulting in creating shield between combustibles and fuels.

Putting out the LIB fire is not only to suppress the fire from LIB but also require an effort to stop the chemical reactions inside LIB [1]. However, liquid submersion extinguishants can both extinguish the fire and cool down the burned surface, but it cannot inhibit LIB chemical reactions which is just prolongs burning process resulting in battery re-ignition. Moreover, the suppression of LIB fire and explosion usually observed using liquid submersion system, but which type of fluid used in liquid submersion system still needs further research information to optimize conditions [24].

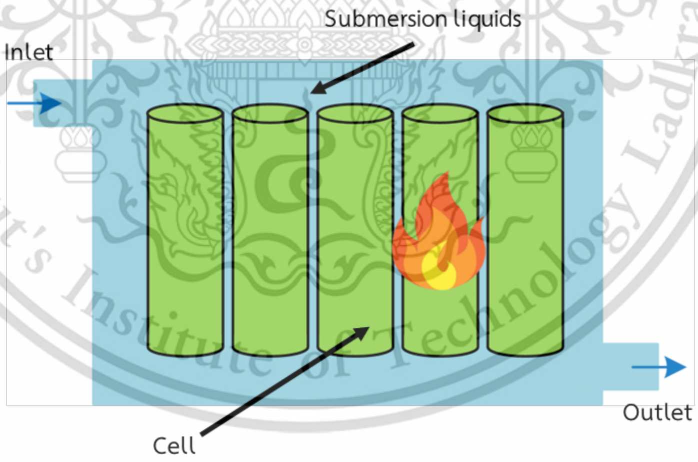


Figure 2.8 Liquid submersion suppression system adapted from [25]

Table 2.2 Electric Vehicle accidents in recent years

No.	Date	Location	Cause	Description	Detail
1	2016.01	Norway	ISC	The Tesla Model S caught fire while fast-charging at Supercharging Station	ISC, Due to the damage to the car, the exact cause of ISC could not be identified [3], [5]
2	2016.04	China	ISC	A Wuzhou Dragon EV bus caught fire	Short circuit caused by wire deterioration [3], [5]
3	2016.06	China	Overheat	An iEV5 caught fire before the landmark of Sanlitun	Might be overheat caused by wire connection loose [3], [5]
4	2016.07	China	ISC	Battery pack of an EV bus caught fire after heavy rain	Water immersion brought ISC [3], [5]
5	2016.07	Italy	Unknown	An BMW i3 BEx EV police car caught fire on the street	Unknown [3], [5]
6	2016.08	France	Unknown	An EV (Tesla Model S) car caught fire during a promotional tour	Unknown [5]
7	2017.03	China	Overcharge	The Tesla EV caught fire during charging at a supercharging station	Overcharging [2]
8	2017.08	USA	Crash	Tesla Model X caught fire after crashing into a garage	Crash deformed the battery modules initiating ISC and fire [5]

Table 2.2 Electric Vehicle accidents in recent years (Cont.)

No.	Date	Location	Cause	Description	Detail
9	2018.01	China	Battery defect	The Tesla EV caught fire during storage	Battery safety system design defect [2]
10	2018.01	China	Unknown	The Tesla Model S caught fire in the parked vehicle	The car had supercharged just hours before the fire [20]
11	2018.03	Thailand	Overcharge	Porsche Panamera caught fire while being charged	Overcharging [20]
12	2018.03	Spain	Unknown	BMW i3 Rex caught fire in the parked vehicle	Unknown [20]
13	2018.03	USA	Crash	The Tesla Model X caught fire after crash	ISC, Fire extinguished in the scene but reignited twice at tow yard 5 days later [20]
14	2018.05	USA	Crash	The Tesla Model S caught fire after crash	ISC, Fire initially extinguished quickly but reignited during loading on tow truck and once again at tow yard [20]
15	2018.05	Switzerland	Crash	The Tesla EV caught fire after crash	Crash [20]
16	2018.05	China	Self-ignite	The Zhong Tai EV caught fire while being driven	Self-ignite without traffic accident [20]

Table 2.2 Electric Vehicle accidents in recent years (Cont.)

No.	Date	Location	Cause	Description	Detail
17	2018.06	USA	Unknown	The Tesla Model S caught fire while being driven	Fire extinguished on the scene without reignition [20]
18	2018.12	USA	Unknown	The Tesla Model S caught fire in the parked vehicle	Fire reignited twice [20]
19	2019.01	USA	ISC	The Tesla Model S caught fire after crash	Crash [5]
20	2019.01	Australia	ISC	Electric road bike exploded and caught fire	ISC [5]
21	2019.03	Netherland	Unknown	The BMW i8 plug-in hybrid started to smoke in a showroom	Unknown [5]
22	2019.05	China	Overcharge	Dongfeng EV truck caught fire after charging	BMS failure inducement overcharge [2]
23	2019.10	Norway	Overheat	Hybrid ferry "Ytterøyningen" caught fire and explosion	Fire started somewhere else and then spread to parts of the battery compartment [7]
24	2020.10	Worldwide	Battery defect	BMW plug-in hybrid battery heated caught fire	Impurity in the battery got in the battery during production process [2]
25	2021.03	Norway	Overheat	Hybrid-electric tour vessel boat catches fire	Fire broke out in or near stand board battery compartment [8]

Table 2.3 Electric vehicle accidents using water-based fire suppression

No.	Date	Location	Cause	Incident	Fire extinguishing
1	2017.07	USA	Crash	Tesla Model S crashed into Lake Forest Home igniting fire	Used water as fire extinguishants [26]
2	2017.10	Austria	Crash	Tesla Model S crashed in Austria and turns into fire	Used water as fire extinguishants [27]
3	2018.03	USA	Crash	The Tesla Model X caught fire after crash accident using Tesla Autopilot	200 gallons of water and foam, re-ignition occurred 5 days after crash [28]
4	2018.05	USA	Crash	The Tesla Model S caught fire after crash accident	200-300 gallons of water and foam, re-ignition occurred twice after fire extinguished [29]
5	2018.06	USA	Unknown	The Tesla Model S caught fire while driving, no re-ignition occurs after fire extinguished	Almost 300 gallons of water and foam [30]
6	2018.12	USA	Unknown	Brand new Tesla car burst into fire when parked in workshop	2,000 gallons of water, caught fire three times in a day [31]
7	2019.02	USA	Crash	Fiery Tesla Model X smashes head-on into a tree, the vehicle caught fire and suffer extensive damage	3,000 gallons of water [32]
8	2019.03	Netherlands	Unknown	BMW i8 caught fire in a Netherlands dealership, smoke coming from vehicle in the showroom	Drop the car into a container filled with water for 24 hours [33]

Table 2.3 Electric vehicle accidents using water-based fire suppression (Cont.)

No.	Date	Location	Cause	Incident	Fire extinguishing
9	2019.10	Austria	Crash	Tesla vehicle crashed into a tree then caught fire after first hitting	11 tons of water and special container [34]
10	2019.10	USA	Crash	Tesla Model X SUV caught on fire after a head-on crash	Fire retardant foam [35]
11	2020.04	Taiwan	Crash	Tesla Model 3 caught fire after hitting a traffic island and overturning	Used water as fire extinguishants [36]
12	2021.07	China	Crash	Tesla electric car caught fire following a crash	20 tons of water [37]

Table 2.4 Details and effectiveness of LIB fire extinguishants [5], [21], [22]

Types of Extinguishants	Details	Effectiveness
Water-based	- Most cost-effective extinguishant - Water can be react with $LiPF_6$ and form toxic and harmful HF - Produce combustible hydrogen - Caused external short circuit in cell - Used large amount of water	- Successfully extinguish EV fire - Gas and smoke still generate after waste terminated - Re-ignition still occurs after extinguished by water
Foam	- Cool and seal surface putting barrier between flammable vapour and hot surface/fuel and starving the fire of air	- Extinguishes fire faster than water - Cooling effect is less effective than water extinguishant
Powder/Dry powder	- Not provide cooling - ABC powder can prevent re-ignition for different types of LIB	- Surface temperature is still high caused surrounding LIB overheated - Suitable for single
CO_2	- Smoothing the fire by replacing O_2 with CO_2 - Not an ideal LIB fire suppressant due to its low cooling capability	- Cannot cool the battery down due to its low heat capacity - Re-ignition is often observed

Table 2.4 Details and effectiveness of LIB fire extinguishants [10]–[12] (Cont.)

Types of Extinguishants	Details	Effectiveness
Halon-based	- Leave any residue - Eliminates radicals necessary to maintain combustion, extinguishes fire by interrupting chain reactions	- Could put out LIB fire but re-ignition might occur
HFC-227ea	- Decreasing oxygen concentration by gaseous agent	- Rapidly put out LIB fire within several seconds - Extinguished fire burns again after the agent terminated
$C_6F_{12}O$ (Novec 1230)	- Vaporization easily occurs due to its low boiling point which could carry a lot of heat away	- Control LIB fire within 30 seconds - Continuous spray on battery surface is necessary to prevent re-ignition
Aerosol	- Low environmental impact, low toxicity, non-conductive and low residue	- Rapidly extinguish LIB fire in closed places - Re-ignition still biggest challenge

Table 2.5 List of types of water-based fire extinguishants

Types of water-based	Details	Effectiveness
Water jet	- Apply a stream of water directly to burning LIB	- Provide cooling and inhibiting re-ignition - Has electric shock concerns
Water spray or sprinkler	- Used a spray of fine water droplets (above 1000 um) - Has enough momentum to penetrate the fire plume and cool surface	- Effectively reduce maximum temperature - Ineffective in compartment system because of difficulty to penetrate inside the battery - Does not prevent the propagation of TR, but delays the onset temperature
Water with added surfactants	- Improve the efficiency of water extinguishment - Decreasing water's surface tension	- Coat the burning materials and cools more efficiency
Water mist	- Droplet size under 1000 um - Less water consumption, low cost, no toxic, low electrical conduction and environment-friendly - Greater absorption of heat energy due to larger surface area to volume ratio - Cooling and oxygen dilution play a dominant role	- Efficiency subjected to spray moment, spray time and water volume - Effectively prevent TR propagation in multi-batteries - The uniformity of WM, hardly reach the surface of LIB and easily affected by ventilation condition are main problems - Problem of application in battery module scale

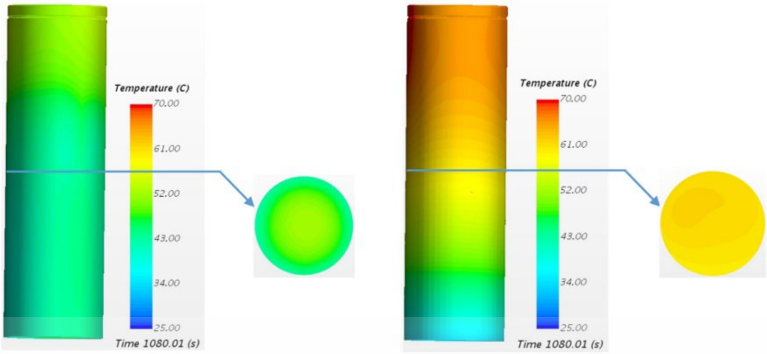
Table 2.5 List of types of water-based fire extinguishants (Cont.)

Types of water-based	Details	Effectiveness
Liquid submersion	- Submerging full coverage of the cell into liquids directly - Usually observed using liquid submersion system	- Providing temperature uniformity and safely maintains target temperature - Preventing thermal propagation of the TR cell to adjacent cell

2.5 LITHIUM-ION BATTERY LIQUID SUBMERSION

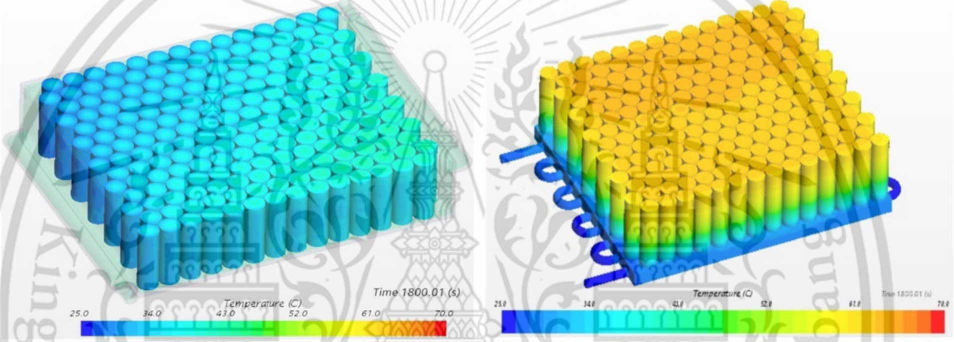
Shaw-Stewart et. Al. [38] conducted 24 hours cell discharging of 100% SoC LIB cells in 5 wt.% electrolyte solutions. The results show that 5 wt.% NaCl solution had the highest corrosion rate on the electrode of LIB, but simultaneously provided good discharge performance. The positive terminal of LIB corroded fast and barely lasted for 1 hour. However, stable voltage cannot be measured throughout the experiment and ended up with a toxic liquid waste due to the leakage out of battery electrolyte. On the other hand, many salt solutions show slight discharge without serious corrosion. There was no fire and explosion observed in discharge experiments according to the hydrogen will not combust unless there is at least 4 vol% of hydrogen in the gaseous mix. Xiao et. Al. [39] perform LIB discharging experiment in 250 mL salt solutions by submerging LIB in different prepared solutions. They found that over 60% of electricity is released within 1 hour and serious corrosion occurs when discharged in concentrated NaCl solutions. Meanwhile, the galvanic corrosion at the positive terminal results in rapid release of electricity. Also, the evidence shows that the positive terminal of battery was destroyed, leading to leakage of organic electrolyte, and forming of toxic liquid waste. However, there was no fire observed after the submersion experiments.

Pulugundla et Al. [10] performed submerged-cell cooling compared to bottom-cold-plate cooling experiments. A single 21700 cylindrical cell is discharged at 3C from 4.2 V to 2.5 V and is submerged into dielectric fluid almost 90% of its height in enclosure box with inlet and outlet channel for dielectric fluid flow. The results show an improved performance in submerged-cell cooling experiment because of higher heat transfer surface area compared to high vertical temperature gradient obtained from bottom-cold-plate cooling experiment as shown in Figure 2.9. Moreover, Dubey et Al. [12] also performed immersion-cooled battery module compared to the cold-plate-cooled module. In immersion-cooled battery module, 196 cells of NCA based cylindrical 21700 Li-ion batteries are used in the enclosure module with one inlet and outlet channel for dielectric fluid flow. All 196 cells are submerged almost 90% of their height to perform immersion-cooled battery module experiment. The results show an improved thermal performance for EV batteries module operated on high C-rate as shown in Figure 2.10. On the other hand, the cell-to-cell temperature difference is still higher than the cold-plate based cooled module experiment.



(a) (b)

Figure 2.9 Temperature contours on the outer surface of the Li-ion cell for (a) submerged-cell cooling (b) bottom-cold-plate cooling from [10]



(a) (b)

Figure 2.10 Temperature contours battery module (a) immersion-cooled battery module (b) cold-plate-cooled module from [12]

CHAPTER 3

RESEARCH METHODOLOGY

3.1 CYLINDRICAL LITHIUM-ION BATTERY

The cell used in these experiments is commercial 18650 Lithium-ion cells with NMC ($LiNiMnCoO_2$) as a cathode of the cell. The same type of TORIYAMA N18650CL-29 Lithium-ion cell was used throughout experiments as shown in Figure 3.1 (a). All of the cells were charged to 4.2 V following to standard charge method, which is CC-CV method, from previous report [40]. In charging process, the R&S®HMP4000 power supply and FLIR E8 Thermal Imaging Camera were used to apply CC-CV method and monitor temperature of cell throughout charging process as shown in Figure 3.1 (b) and Figure 3.1 (c). Moreover, cell resistance and cell weight were measured before experiments using HIOKI BT3554 BATTERY TESTER and METTLER TOLEDO Mettler ML-series respectively as shown in Figure 3.2. Cell specification is shown in Table 3.1.

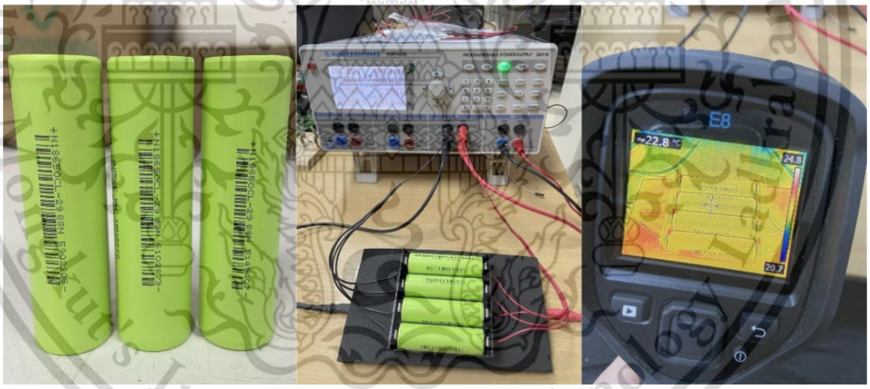
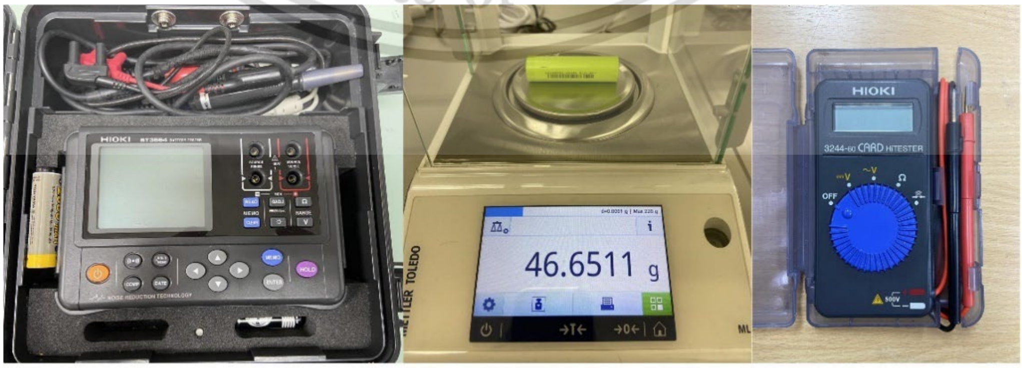


Figure 3.1 TORIYAMA N18650CL-29, charging process and thermal imaging camera



(a) (b) (c)

Figure 3.2 (a) Hioki BT3554 battery tester (b) Mettler Toledo ML-series and (c) Hioki 3280-10F

3.2 SUBMERGED LIQUIDS

The experiment was performed using several types of liquid including Natural seawater (NSW), Synthetic seawater (SSW), single salt solutions from SSW, DI water, and Thai tap water. The NSW was collected from Bangsaen Beach in Chonburi province, Thailand. The SSW was prepared by dissolving 35.675 grams of the main sea-salt in DI water with enough DI water added to make one-liter total solution. The single salt solutions included NaCl, $MgCl_2$, Na_2SO_4, $CaCl_2$ and KCl solutions. Thai tap water was collected from Pathum Thani province in Thailand. In this experiment, only 500 ml. of each solutions/liquid were used to submerge the cell in each experiment. In addition, the main sea-salt of SSW used in the experiment is shown in Table 3.2 [41].

Table 3.1 TORIYAMA N18650CL-29 Cell specification [40]

ITEM	SPECIFICATION
Nominal capacity [mAh]	2900 @0.2C
Nominal voltage [V]	3.6
Charge voltage [V]	4.2
Discharge cut-off voltage [V]	2.5
Cell dimension [mm]	Height: 64.85± 0.25 Diameter: 18.35± 0.15
Weight [g]	≤ 48
Standard charge method	CC-CV, standardly 1375 mA, 4.2V (28 mA)

Table 3.2 Main chemical composition of synthetic seawater [41]

COMPOUND	CONCENTRATION [g/L]
NaCl	24.53
$MgCl_2$	5.20
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.695
Total	**35.675**

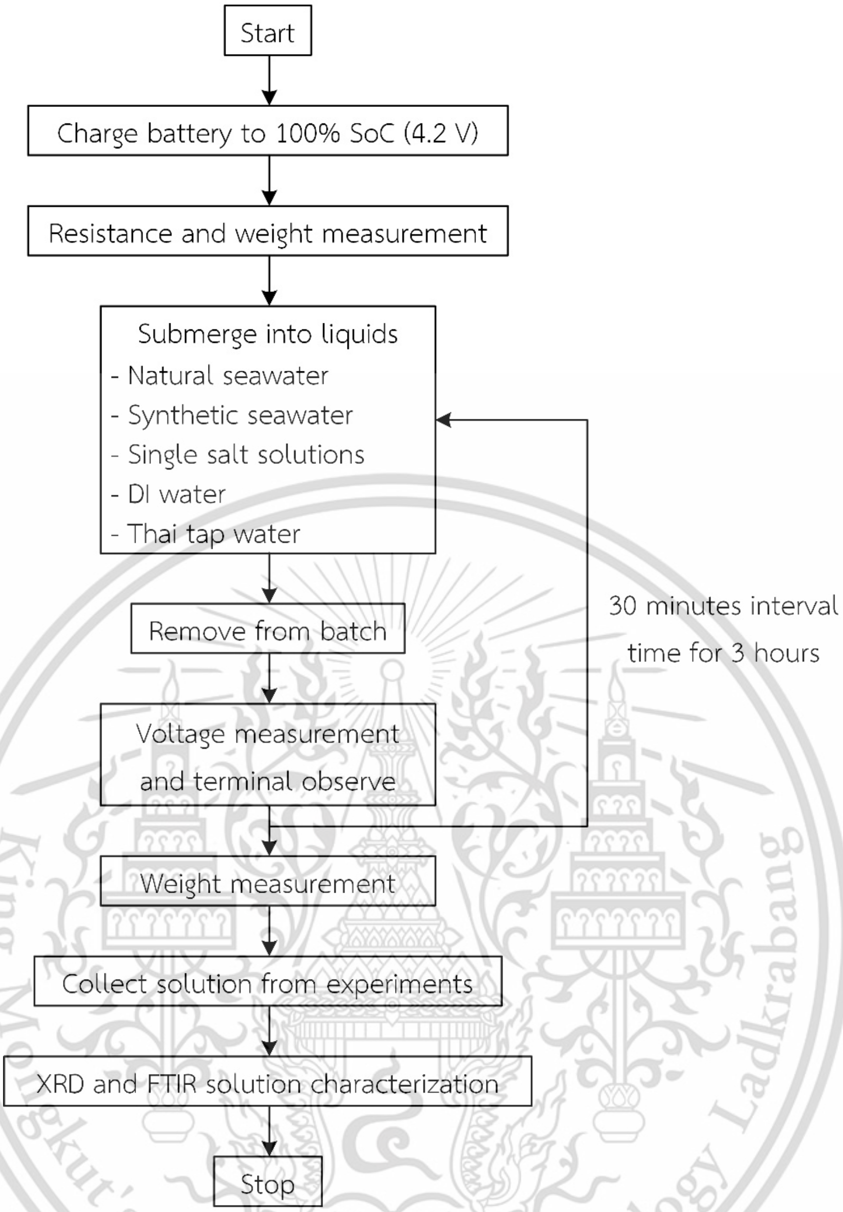


Figure 3.3 Methodology for Lithium-ion battery liquid submersion experiment

3.3 LITHIUM-ION BATTERY LIQUIDS SUBMERSION EXPERIMENT

After the cells were charged up to 4.2 V (100% SOC) following to CC-CV method from Table 3.1, each cell was submerged into liquid batch, to start the liquid submersion experiments. The NSW was used to validate the use of SSW in subsequent submersion experiments. The voltage of the cell was measured, and the solutions during/after submersion were observed to compare the discharge rate and reactions of NSW and SSW submersion. Then, the single salt solutions were used to analyze the effect of specific salt solutions on discharge rate and characteristic of solutions after submersion experiment compared to SSW. The overall methodology of liquids submersion experiments is shown in Figure 3.3. Then, after SSW submersion, the sediment in covered liquid were characterized out by XRD (Rigaku - TTRAX III X-ray diffractometer) and FTIR (Nicolet™ iS50R Research FTIR Spectrometer) with 7800–350 cm^{-1} spectral range for standard system to investigate products from submersion experiments and electrolyte organic leakage during/after SSW submersion respectively as shown in Figure 3.4. Each cell was removed from the liquid 30 minutes intervals time to measure cell's voltage using handheld multimeter (HIOKI 3280-10F), observe terminal corrosion physically, and take a picture each terminal of the battery cell then removed from liquid after 3 hours submersion as shown in Figure 3.5. Moreover, the magnetic stirrer was applied to SSW submersion experiments to simulate circulated-liquid submersion fire suppression system for future application that has a possibility to apply into EV.

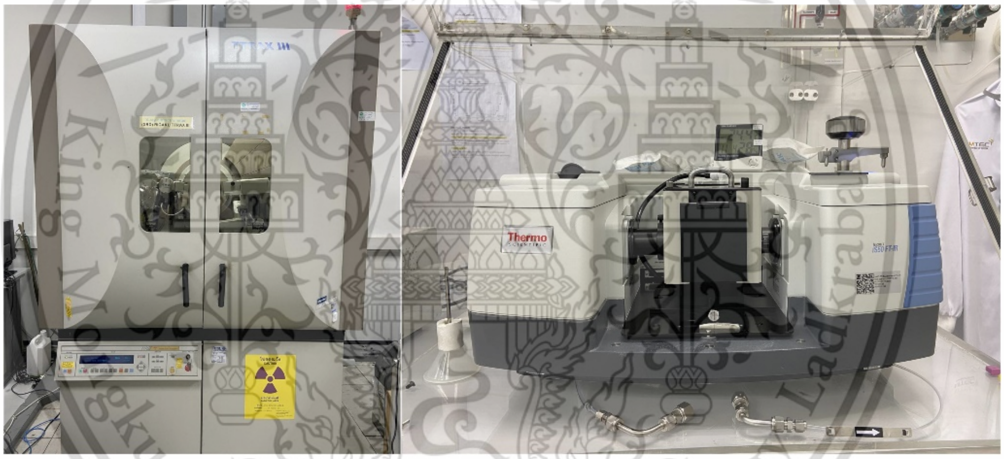

(a) (b)

Figure 3.4 (a) Rigaku-TTRAX III X-ray diffractometer and (b) Nicolet™ iS50R Research FTIR Spectrometer

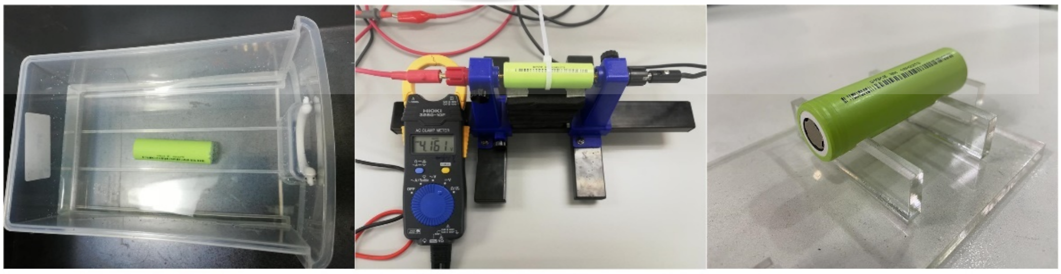

(a) (b) (c)

Figure 3.5 Liquids submersion experiment (a) Single cell submersion (b) Voltage measurement using Hioki 3280-10F handheld multimeter and (c) Terminal corrosion physical observing

3.4 LITHIUM-ION BATTERY THERMAL RUNAWAY PREVENTION EXPERMENT

The experiment was started by using overheated abuse conditions to investigate TORIYAMA N18650CL-29 critical temperature to trigger TR. The 1500 W heater pad was used to provide external heat to fully charged LIB in experiment as shown in experimental schematic and experimental setup in Figure 3.6 and Figure 3.7 respectively with the thermocouple locations and voltage measurement as shown in Figure 3.8.

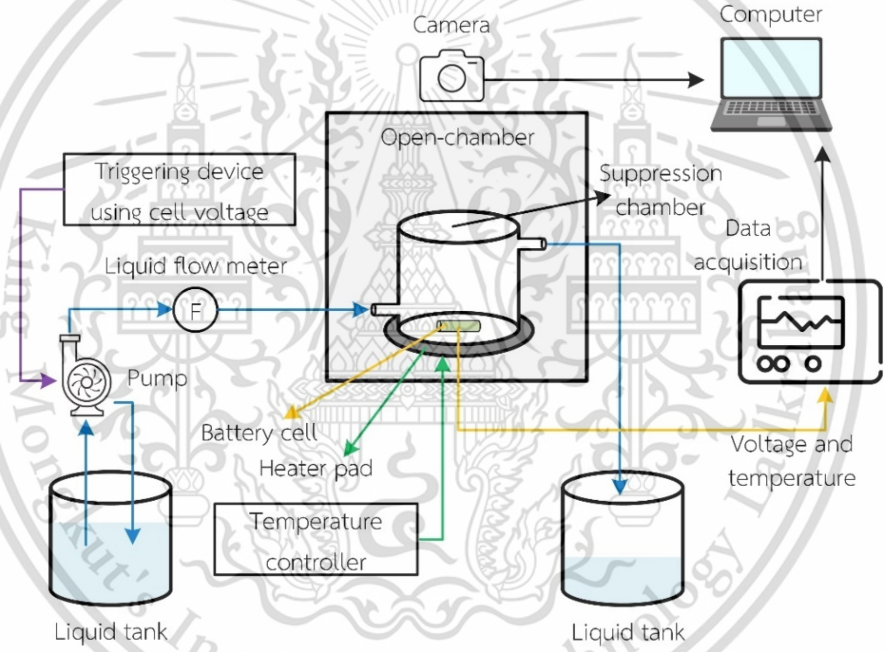


Figure 3.6 Experimental schematic of battery fire suppression experiment

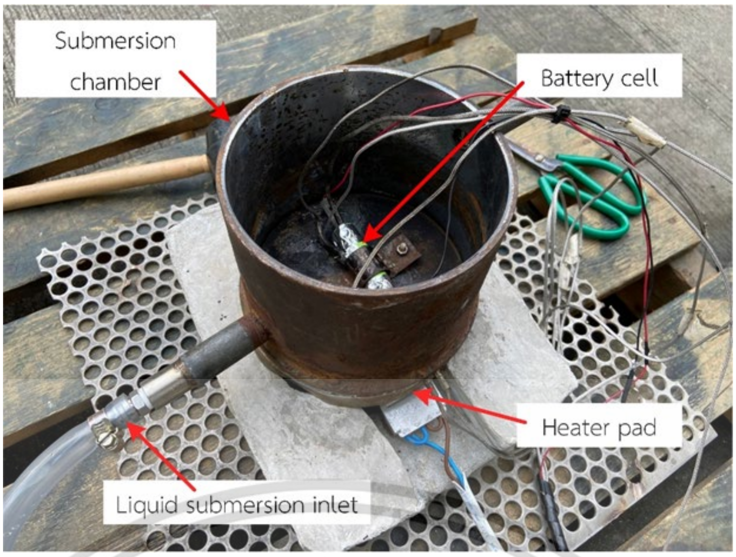


Figure 3.7 Experimental setup of suppression chamber and battery location

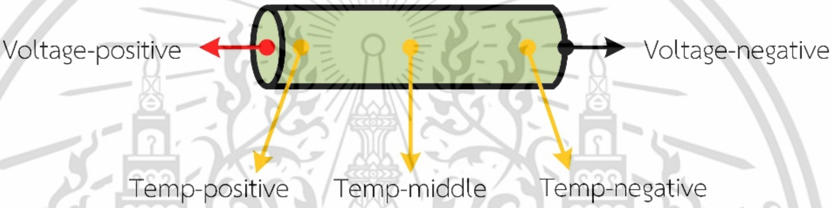
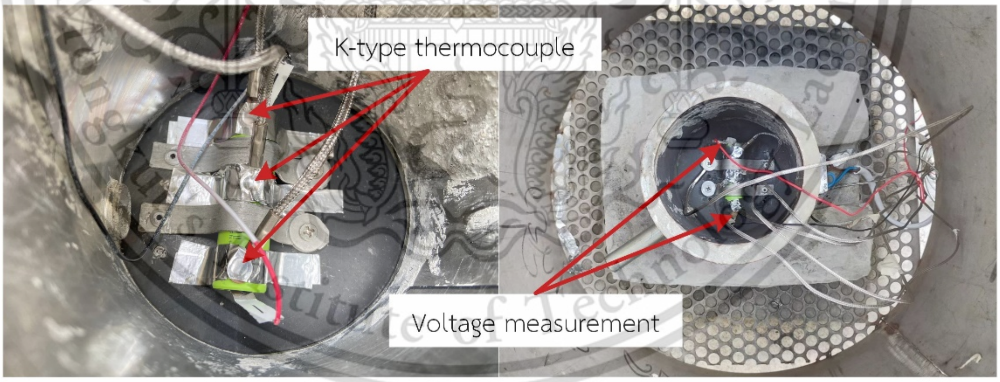


Figure 3.8 Schematic of thermocouple locations and voltage measurement locations for battery fire suppression experiments



(a) (b)

Figure 3.9 Thermocouple and voltage measurement locations setup of battery fire suppression experiment

The PID temperature controller (MAXTHERMO MC-5638) was used to control temperature ramp rate and temperature of heater pad. For temperature measurement, K-type thermocouples and J-type thermocouples were attached to LIB and heater pad then connected to data acquisition (GRAPHTEC midi LOGGER GL 840) with millisecond accuracy. During the experiment, voltage and surface temperature of cell were measured throughout experiment using 200 milli-second sampling rates. Temperature of cell was measured in 3 positions which are positive terminal, middle, and negative terminal as shown in Figure 3.9. Moreover, the behavior of cell was recorded as video entire experiment to observe the behavior of cell at any critical temperature. Then, after critical temperature, voltage, and behavior of cell during subjecting external heat were collected, LIB fire suppression using liquid-submerged technique was conducted to investigate the possibility of using liquid submersion to suppress fire and explosion from LIB. the liquid-submerged system was triggered by using triggering device as shown in Figure 3.10. The triggering device was built by using a single-board microcontroller Arduino UNO R3 with high- and low-level trigger relay 5V 30A and LabVIEW software to control liquid suppression system. During the experiment, the voltage of battery cell was collected using analog input channel of Arduino UNO R3 and the 5V relay was triggered by the voltage drop of battery cell then the normally open (NO) contact and normally closed (NC) contact of relay will be used to turn on liquid suppression system and turn off heater pad respectively.

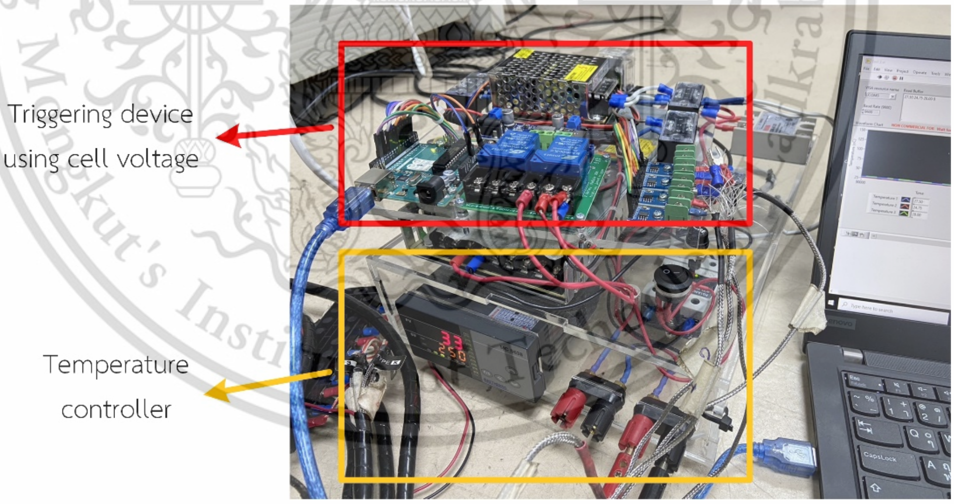


Figure 3.10 Triggering device using cell voltage for liquid submersion and temperature controller for controlling temperature of heater pad

CHAPTER 4

LIQUID SUBMERSION EXPERIMENT

4.1 SYNTHETIC SEAWATER VALIDATION

4.1.1 Voltage of cell discharged in NSW and SSW

Figure 4.1 shows voltage of the cell as a function of time in NSW and SSW submersion experiment. The voltage of the cell submerged in NSW and SSW are shown in dash line and dark-solid line respectively. After submerged cell into NSW and SSW for 2 hours and remove at 30 minutes interval time for voltage measurement, the result shows that voltage of cell and discharge rate of SSW is almost the same compared to cell submerged in NSW. However, the maximum voltage difference is 0.2% at 2 hours submersion time which can be considered as no statistically significant difference. This is probably because of the difference of solution concentration and salt compositions between NSW and SSW. Nevertheless, the positive terminal of cell submerged into seawater is corroded and destroyed in 2 hours submersion time resulting in the voltage of the cell cannot be measured and the termination of this experiment. Moreover, no fire and explosion observed after this liquid submersion experiment.

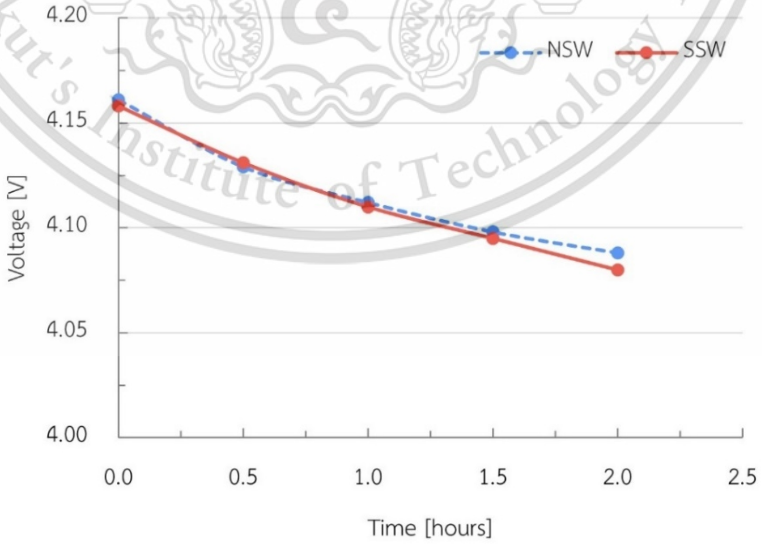


Figure 4.1 Voltage as a function of time for NSW and SSW submersion

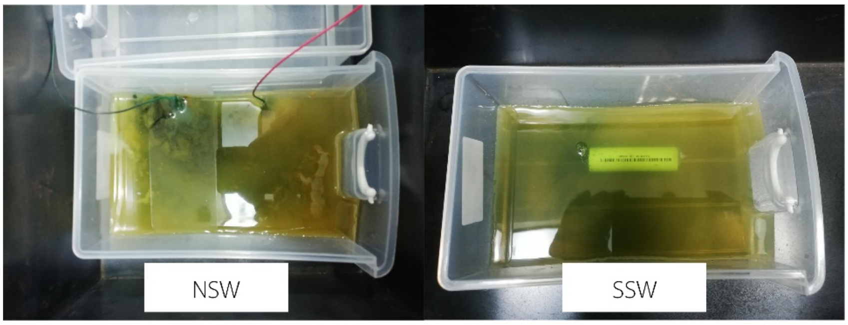


Figure 4.2 Solutions after submersion experiment

4.1.2 Solution after experiment

During NSW and SSW submersion experiment, the solutions are turned to greenish tinge. This is probably Ferrous hydroxide ($FeOH_2$) from the product of galvanic reactions as show in Equation (4.1) and Equation (4.2) [39]. Then, the solution turned yellow and brown during NSW and SSW submersion. Due to the collapse of positive terminal, an organic electrolyte was probably dissolved into solution making solution from submersion seriously contaminated. That is the reason why the solution characterizations are used in this research. It will be discussed the detail in later topics. Moreover, from comparing discharge rate and solution during/after submersion, SSW has an ability to be used as NSW because NSW contains many kinds of salt.

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \tag{4.1}$$

$$Fe^{2+} + 2OH^- \rightarrow FeOH_2 \tag{4.2}$$

4.2 SINGLE SALT SOLUTIONS

Due to the positive terminal corrosion of the cell submerged in SSW, single salt solutions with the same salinity as composition of SSW from ASTM D1141-98 standard [41] are used to analyze the effect of salt solutions on terminal corrosion. The main chemical compositions of SSW used in this experiment are Sodium chloride (NaCl), Magnesium chloride ($MgCl_2$), Sodium sulfate (Na_2SO_4), Calcium chloride ($CaCl_2$), and Potassium chloride (KCl) as shown in Table 3.2.

In this experiment, each LIB cell was submerged into SSW and single salt solutions of SSW to analyze and compare the effect of types of salt solution on discharge rate and submerged solutions after submersion experiment.

4.2.1 Voltage and discharge rate

Figure 4.3 shows voltage of cell as a function of time during single salt solutions submersion experiment with the dark-solid line as SSW and dash line as each salt solutions. From the results, there is a marginal voltage discharging in the cell submerged in $MgCl_2$, Na_2SO_4, $CaCl_2$, and KCl because of the amount of $MgCl_2$, Na_2SO_4, $CaCl_2$, and KCl composed in SSW is much lower than NaCl. On the other hand, a rapid discharge occurs on cells submerged in SSW and NaCl solution with the same trending. However, the voltage of cells submerged in SSW is lower than those in NaCl at the termination of experiment. This is probably because the lower concentration of NaCl content dissolved in solutions resulting in the lower electrical conductivity of solution compared to SSW. As a result, it can be concluded that voltage discharge behavior of cell submerged in both NSW and SSW mainly comes from NaCl salt dissolved in solution because of the amount of NaCl is the most dominant salt contained in both NSW and SSW.

4.2.2 Single salt solution after submersion

During submersion the cell in single salt solutions, the galvanic corrosion reactions continuously occurred and generated the product dissolved into solutions. The sediments represented in solution are different in each batch as shown in Figure 4.4. Evidently, sediments produced from submersion in SSW and NaCl are the same as shown by the green, black, and some orange sediment confirming that the terminal corrosion reaction mainly comes from NaCl. However, the products generated in $MgCl_2$ batch are almost the same, but a smaller number of sediments dissolved as in SSW and NaCl batch. Due to the less amount of $MgCl_2$ dissolved in solution compared to NaCl, the amount of product generated from galvanic reaction at positive terminal of the cell is lower. This solution after submersion can denote that chloride-containing anions in solution is one of the reasons that galvanic corrosion occurred at the positive terminal of the cell. In addition, the orange sediment represents in $CaCl_2$ and KCl batch probably causes by chloride anions in composition. Furthermore, Na_2SO_4 batch produced a small amount of green and orange sediments dissolved in solutions.

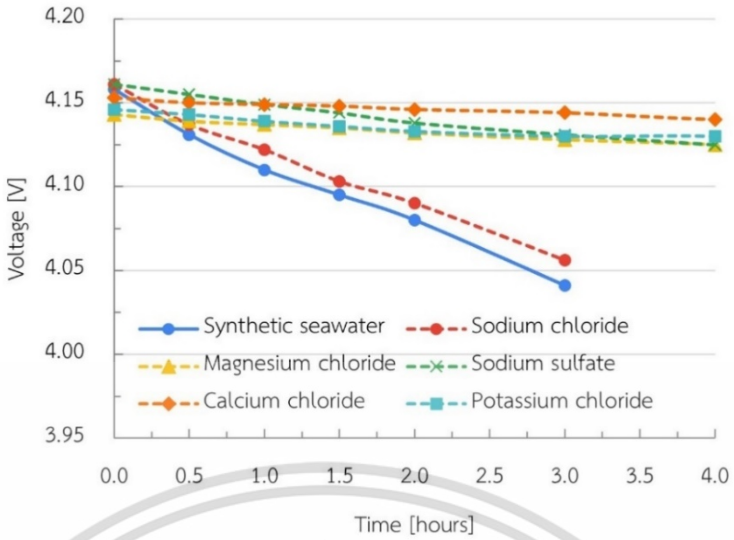


Figure 4.3 Voltage as a function of time for cell submerged in SSW and single salt solutions from main components of SSW

Figure 4.4 Solutions after single salt solutions experiment

4.2.3 Terminal corrosion and deposition

Positive terminal corrosion and negative terminal decomposition of the cell are shown in Figure 4.5. The results indicate that serious terminal corrosion occurs on positive terminal of cell submerged in SSW and NaCl. Also, minor positive terminal corrosion occurs on the other salt solutions because of less concentration than SSW and NaCl solution. Moreover, on negative terminal surface, there is apparently salt deposition in some cases which are SSW and $MgCl_2$ due to its competing reaction. Thus, it can be concluded that the primary cause of serious positive terminal corrosion comes from the competing reaction of NaCl, which is main composition of both natural NSW and SSW.

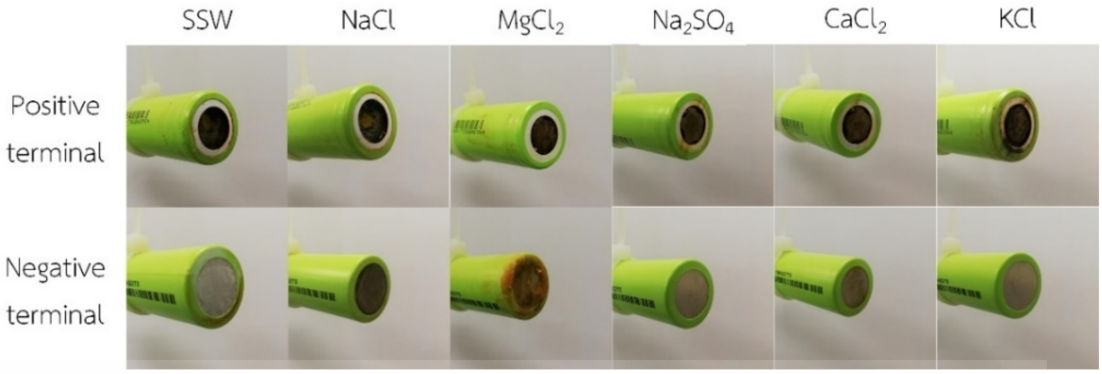


Figure 4.5 Terminal corrosion and deposition of single salt solutions submersion

4.3 VARIOUS TYPES OF LIQUID SUBMERSION

In this experiment, SSW, DI water and Thai tap water were used as a submerged liquid. All submerged liquids are expected that it can be applied to LIB liquid submersion fire suppression system in EV. SSW has the potential to discharge voltage of submerged cell and reduce the risk of reignition after TR, but it had to deal with liquid waste problem after submersion. DI water is technically the most suitable for LIB liquid submersion. Thai tap water is the most suitable in practical use for fire suppression.

4.3.1 Voltage and discharge rate

The cell voltage variation through immersion time is shown in Figure 4.6. The results indicate slight reduction of battery voltage in DI water and Thai tap water. On the other hand, SSW provided rapid discharge behavior to 4.05 V The voltage of cell could not be measured after 3 hours submersion time as shown in Figure 4.6. Normally, submersion into electrolyte solution like seawater discharge energy inside LIB. However, the termination of voltage measurement in this case could be interpreted that vent disk or positive terminal was completely corroded as shown in Figure 4.7, implying that battery was not fully discharged and still stored energy inside. Though, there was no fire and explosion observed after submerging LIB into liquid in these experiments, which confirms that this liquid submersion technique has potential to be used as LIB fire suppression.

4.3.2 Terminal corrosion

For SSW submersion, the top cap of positive terminal, which mainly made of steel, was corroded after 1.5 hours submersion time as shown in Figure 4.9. This was because Cl^- anions in SSW could facilitate metals dissolution, generate flocs resulting in serious galvanic corrosion on positive terminal [39] and the gas production, which is hydrogen gas at negative terminal as shown in Figure 4.8. In addition, the small amount of oxygen and chlorine gas, which is yellow-green gas at room temperature, were produced at positive terminal. Moreover, the cell exhibited black residue on positive terminal surface which is an iron oxide substance. After that, the vent disk of positive terminal was completely corroded in 3 hours submersion time, resulting in the failure of voltage measurement. Moreover, the organic electrolyte inside LIB was possibly leaked out and dissolved into liquid solution after this submersion time. This could be harmful to the environment because Li salt in electrolyte might react with water and form strong acid. On the negative terminal, the deposition of white substance was observed as shown in Figure 4.9. This could be the salt composing of the cations in seawater solution (Na^+, Mg^{2+}, Ca^{2+}, K^+).

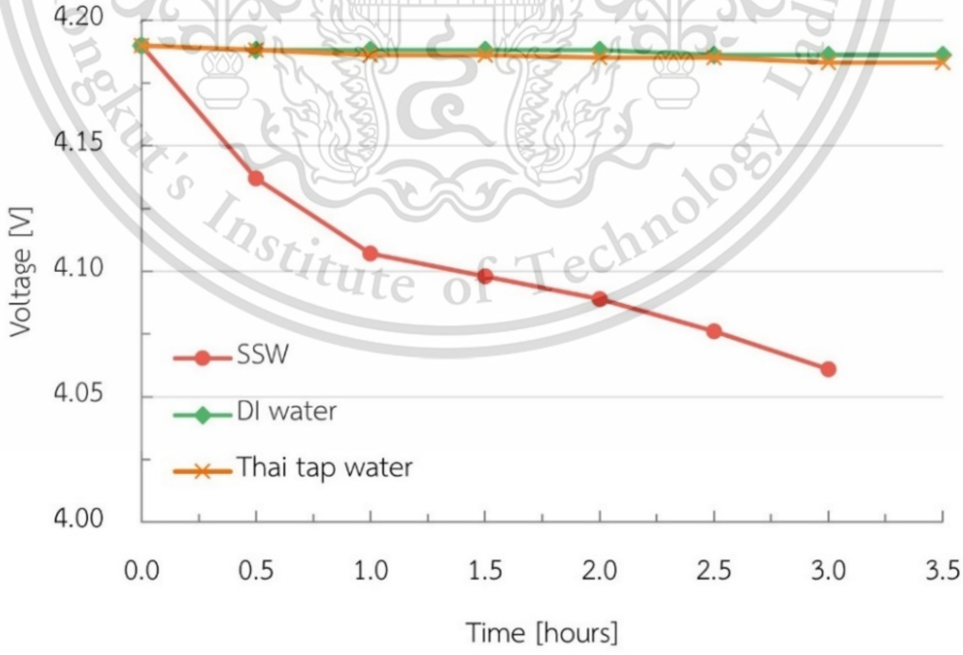


Figure 4.6 Voltage as a function of time for SSW, DI water and Thai tap water

Figure 4.7 Structural collapses of cylindrical LIB during liquid submersion

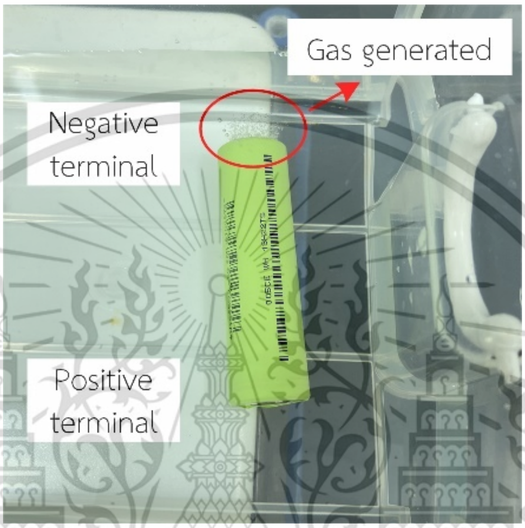


Figure 4.8 Bubbles of gas generated at negative terminal of cell submerged in SSW

As shown in Figure 4.10 (a) DI water seems like an ideal solution for this technique because the serious corrosion occurred on positive terminal of the cell was not observed. For tap water in Figure 4.10 (b), there was small amount of rust on positive terminal and small amount of metal deposited on negative surface due to less some metal impurity and chloride composition in liquid. However, it can be considered as minor terminal corrosion without any harm and toxicity from the leakage of organic substances inside LIB. Therefore, it can be used as practical LIB fire suppressant without fire and explosion.

4.3.3 Weight loss

Table 4.1 shows the cell weight measured before and 1 week after liquid-submerged experiment. The cell in SSW has lost weight around 1.6 grams due to its severe corrosion and terminal deconstruction on positive terminal, and possibly of organic electrolyte inside cell leakage. However, a small weight difference can be observed for the cell in DI water and tap water.

Table 4.1 Weight of cell measured before and after experiment

Liquids	Before	After	Weight
	Weight [g]		change [g]
Synthetic seawater	46.5679	44.9853	-1.5826
DI water	46.8384	46.8353	-0.0031
Tap water	46.6460	46.6483	0.0023

4.3.4 Solution characterization

After 1 week later of experiment, there was organic electrolyte leaked out from LIB at positive terminal of the cell submerged in SSW as shown in Figure 4.11. On the other hand, positive terminal of cell submerged in DI water and Thai tap water was not destroyed and no organic electrolyte leaked out. The leaked substances looked like a gel which can confirm that this type of LIB used gel polymer electrolyte instead of liquid electrolyte. However, there was a risk that a gel polymer electrolyte dissolved into solution. The FTIR characterization was used to identify dissolved organic substances in SSW after submersion experiment.

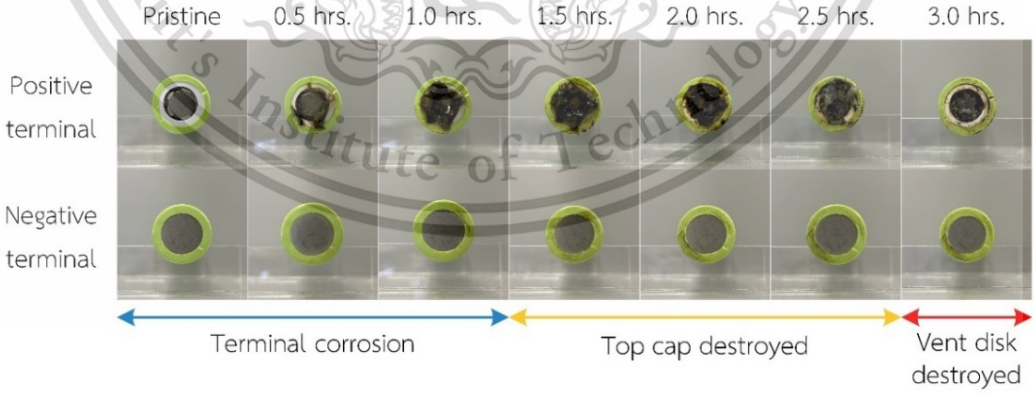


Figure 4.9 Terminal corrosion and deposition throughout SSW submersion

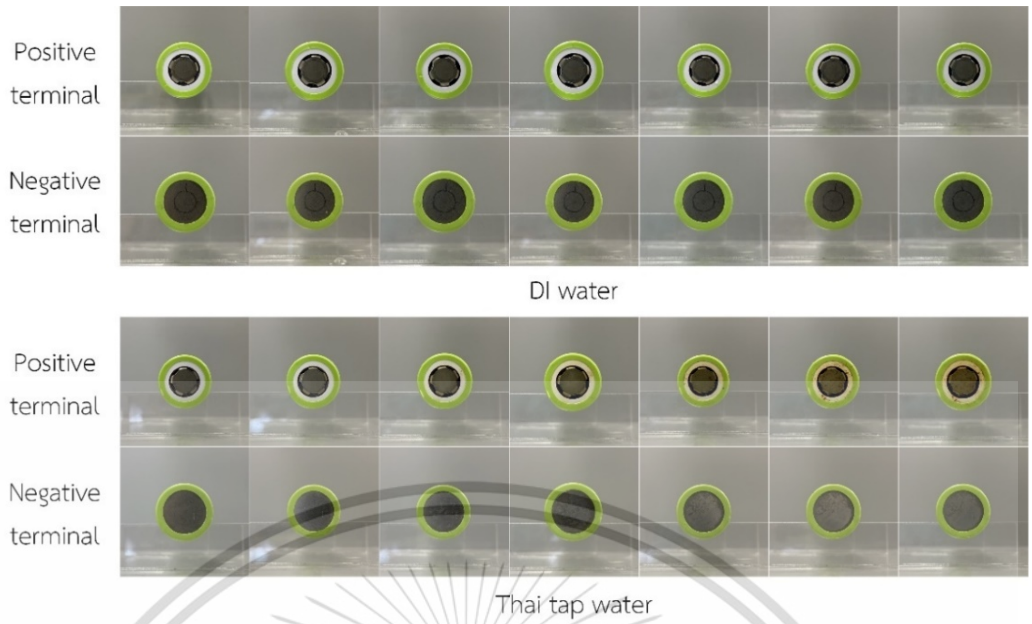


Figure 4.10 Terminal corrosion and deposition throughout DI water and Thai tap water submersion

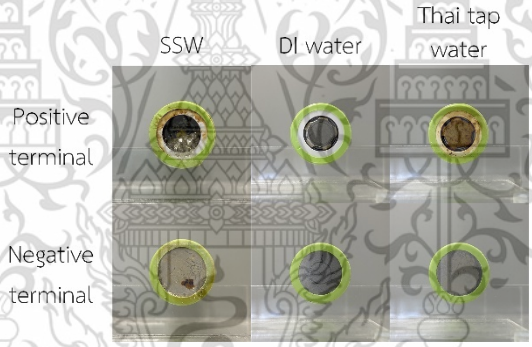


Figure 4.11 Terminal corrosion and deposition 1 week after liquid submersion

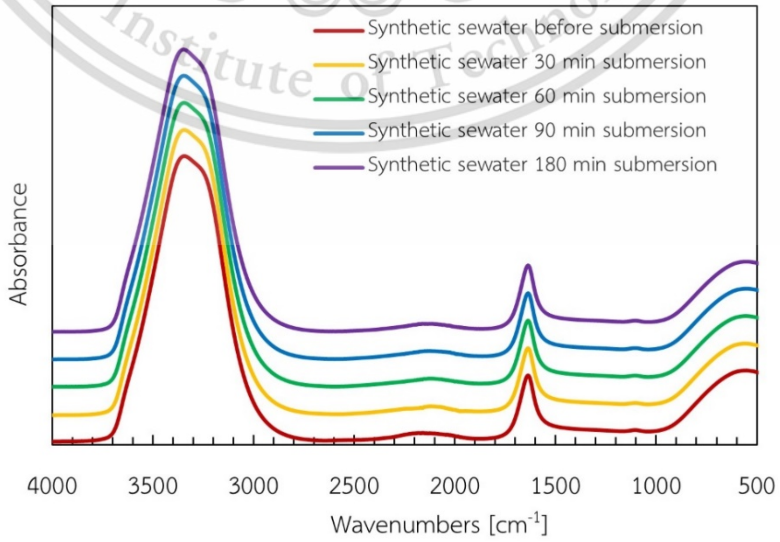


Figure 4.12 FTIR spectra of collected SSW throughout liquid submersion

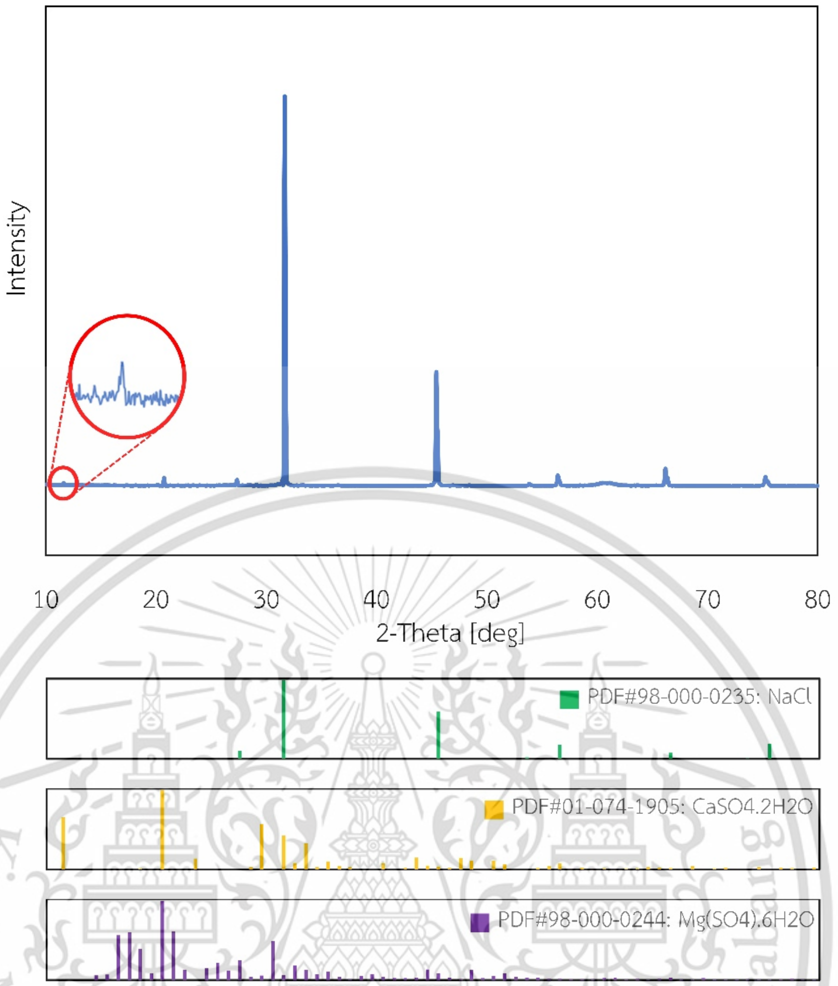


Figure 4.13 XRD pattern analysis of SSW after liquid submersion

The submerged SSW collected from liquid submersion experiment was dried overnight at 80 degree-Celsius and the sediment was collected and characterized. The FTIR characterization result is shown in Figure 4.12. Based on the results of the FTIR characterization, the absence of detectable organic substances in any of the collected solutions suggests that the gel organic electrolyte did not dissolve into the submerged solution during the 3 hours submersion in SSW. While the absence of differences in the FTIR spectra does not conclusively rule out the presence of organic substances, it indicates that FTIR was not sensitive enough to detect any organic substances if they were present. Therefore, the evidence supports the conclusion that the gel organic electrolyte did not dissolve into the submerged solution during the submersion period.

During the submersion experiment, the solution turned yellow and then orange/brown, indicating the generation of iron rust and oxygen compounds of iron due to positive terminal corrosion. However, the XRD pattern presented in Figure 4.13 did not show any presence of iron rust or oxygen compounds. It is possible that the iron and oxygen compounds generated during corrosion dissolved in the SSW and formed amorphous structures, which cannot be detected by XRD.

Therefore, the XRD pattern indicates the presence of NaCl, $CaSO_4$, and $MgSO_4$ in the solution. However, the absence of Fe detection in the XRD pattern highlights the need for additional characterization techniques to accurately identify the presence of Fe in the solution.

Overall, the results suggest that the use of SSW as a fire suppressant poses a liquid waste problem to the environment, as evident from the generated substances from the corrosion of the main composition of LIB positive terminal. Furthermore, it is crucial to limit the submersion time of SSW to 1.5 hours or 90 minutes to prevent serious corrosion from completely destroying the positive terminal and increasing the risk of organic electrolyte leakage.

4.4 THE EFFECT OF STIRRED SOLUTION

After various types of liquid submersion indicating that there was no fire and explosion occurred during/after liquids submersion experiments, single cell was submerged into SSW with magnetic stirrer to simulate liquid-circulate submersion system. In this experiment, each cell was submerged in SSW with magnetic stirrer compared to SSW without magnetic stirrer.

4.4.1 Voltage and discharge rate

Figure 4.14 shows voltage and pH as a function of time on primary axis and secondary axis respectively. The voltage and pH of submersion solution with and without magnetic stirrer are shown in dash line and dark-solid line respectively. The result shows that the cell submerged in SSW with magnetic stirrer has lower voltage than the cell submerged in SSW without magnetic stirrer implying that magnetic stirrer increases discharge rate after 1 hour submersion time. Moreover, cells submerged in non-stirred SSW can last longer than cells submerged in stirred SSW before the termination of submersion due to voltage cannot be measured. This result indicates that circulated-liquid submersion fire suppression system has a possibility to be used as LIB fire suppression system. However, this experiment still needs further information to reconfirm how effective this technique.

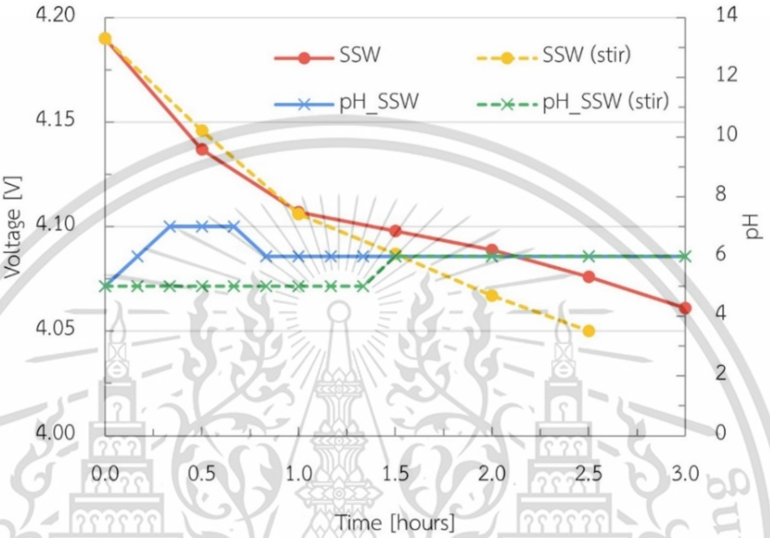
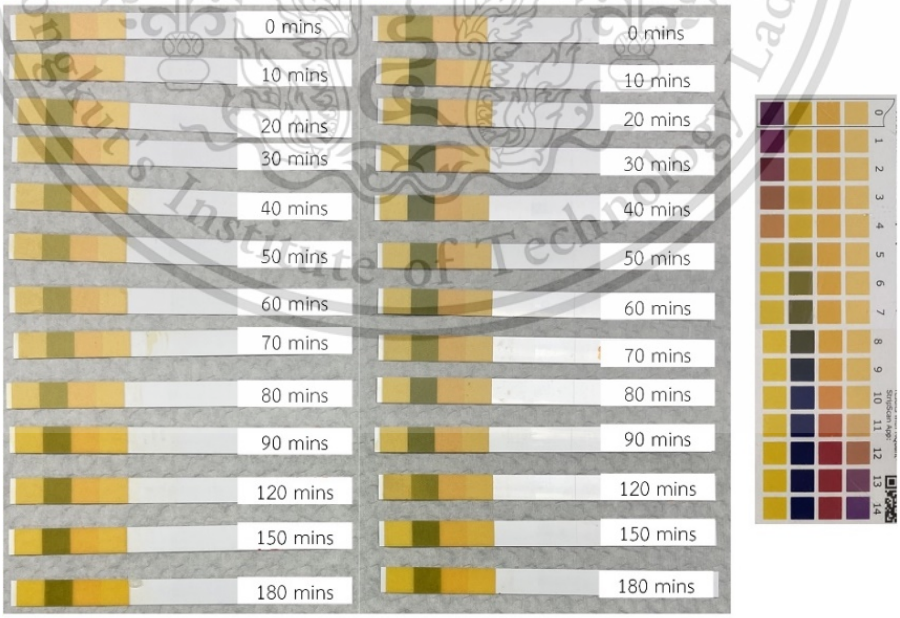


Figure 4.14 Voltage and pH of solutions as a function of time for SSW with and without magnetic stirrer



Stirred solution Non-stirred solution

Figure 4.15 pH of solutions measurement using Litmus paper

4.4.2 pH and solution observing

Because of there was difference of discharge rate between cell submerged in stirred SSW and non-stirred SSW, the pH of submerged solution was measured using Litmus paper as shown in Figure 4.15. Likewise, the values of pH were plotted as a function of time on secondary axis as shown in Figure 4.14. The results show that pH of on-stirred SSW fluctuated. This was probably because of sediments generated during SSW submersion. Figure 4.16 (a) and (b) shows solution after 10 minutes and 3 hours submersion time respectively. After 10 minutes submersion time, the stirred SSW begins to turn orange and brown color. On the other hand, non-stirred SSW was observed that there was no significant product generated making solution turned into orange and brown. However, the on-stirred SSW after 3 hours submersion time as shown in Figure 4.16 (b) appeared to generate green and white sediments dissolved into SSW which are probably an Iron (II) hydroxide ($FeOH_2$) from Equation 4.2 and Aluminum oxide corroded from vent disk which is mainly made of Aluminum. In addition, the Iron (II) ions are easily substituted by Iron (III) resulting in reddish-brown solution in the case of stirred SSW. Surprisingly, after 1 week from the termination of this experiment, non-stirred SSW collected after submersion had changed color from green to reddish-brown same as stirred SSW implying that using stirred solution has increased reactivities resulting in the increasing discharge rate and more serious terminal corrosion. Moreover, the pH and solutions after submersion in this experiment proves that submersion in stirred salt solution like seawater still ended up with liquid waste problem which is the pollution and harmful toward the human health and environment.

4.4.3 Terminal corrosion and deposition

As the terminal corrosion and deposition was observed as shown in Figure 4.17, the result shows that there was the same corrosion characteristic on positive terminal. Comparing to non-stirred SSW terminal corrosion, the only difference of terminal corrosion is the positive terminal was completely destroyed within 2.5 hours submersion time instead of 3 hours submersion time as shown in Figure 4.9 implying that using liquid circulated system can caused more serious terminal corrosion than non-circulated system. This is the reason why the termination of stirred SSW submersion was 2.5 hours. Anyways, using liquid-circulated system as a LIB fire suppression is probably more suitable because of the enhancement of heat transfer rate and heat transfer performance from the moving solution. Moreover, there were no significant deposited substances that appeared on negative terminal same as non-stirred SSW submersion.

(a) (b)

Figure 4.16 Solutions after SSW submersion with and without magnetic stirrer (a) 10 minutes after submersion started (b) 3 hours after submersion started

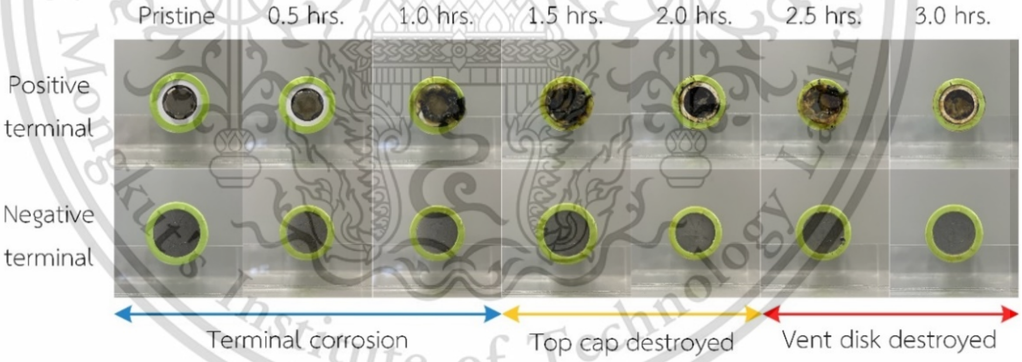


Figure 4.17 Terminal corrosion and deposition of stirred SSW submersion

CHAPTER 5

THERMAL RUNMAWAY PREVENTION EXPERMENT

5.1 CRITICAL TEMPERATURE

To obtain the thermal runaway characteristic of TORIYAMA N18650CL-29 with gel-poly based electrolyte at 100% SOC, the single cell was forced into thermal runaway process by using overheated abuse condition. The cell obtained external heat from heater pad rising to 250 degree-Celsius with ramp rate equal to 3.0 degree-Celsius per minute. Throughout the experiment, the voltage of cell and surface temperature at 3 different locations using type-K thermocouple as shown in Figure 3.9 were measured and recorded. The experiment was carried out until the battery cell experienced a thermal runaway and then exploded. The recorded voltage of cell and surface temperature was shown in Figure 5.1.

The characteristic of thermal runaway can be divided into three stages as shown in Figure 5.1. Stage I, which is the beginning stage of the thermal runaway process, shows the rising temperature with stable voltage of the cell at 100% SOC or 4.2 V. After the rising temperature occurred continuously, battery cell went through the internal short circuit (ISC) at the surface temperature around 97.3 degree-Celsius (Voltage drop temperature: T_d) caused the voltage of cell begins to drop to around 2.5 V. This phenomenon was occurred because of the inside temperature of battery cell had reached the melting point of the separator, which is polymer-based material like polyethylene (PE), causing the short circuit between positive material and negative material inside battery cell.

Stage II happened from the sharply dropped voltage to the unexpected rise of battery temperature. During this stage, the temperature of battery cell was rising continuously until the surface temperature at middle location of cell obtained 129.7 degree-Celsius then the rupture of safety valve at positive terminal occurred with ticking sound as shown in Figure 5.2. The safety valve rupture was triggered by gas generations from side reactions inside the battery cell which increases internal pressure of battery cell to the level that safety valve of battery at positive terminal opened.

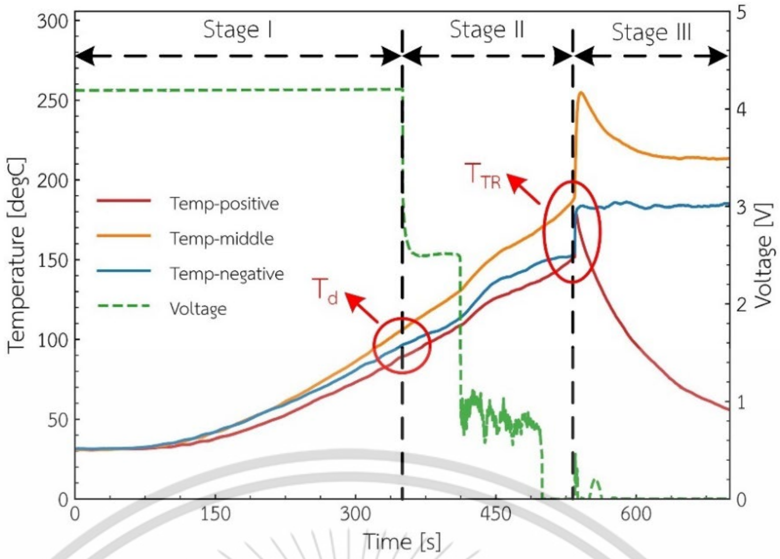


Figure 5.1 Voltage and surface temperature as a function of time entire overheated abuse condition without cooling and suppression

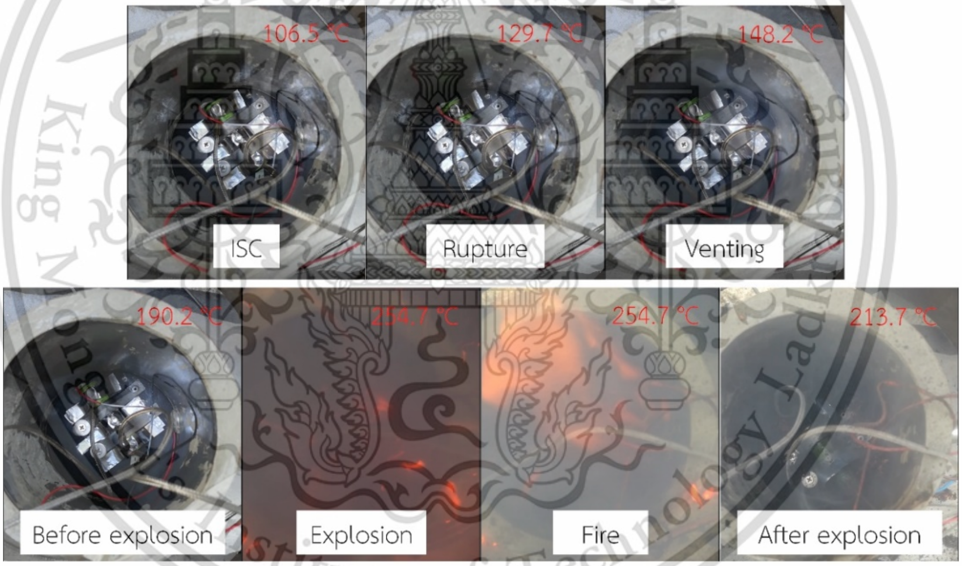


Figure 5.2 LIB exploding process during subjected to overheated abuse condition

After the rupture occurred at positive terminal, the measured temperature was increased significantly with sudden drop of voltage from around 2.5 V to fluctuation up and down between 0.5 V and 1.0 V. This voltage fluctuation is probably because the decomposition of solid electrolyte interface (SEI) and the reactions between electrolyte and negative electrode then forming the regeneration of SEI layer which is important roles to protect open-circuit voltage. In addition, after the rupture occurred, generated gas from side reactions inside battery cell was vented because of the safety pressure relief valve at positive terminal already opened as shown in Figure 5.2.

However, the temperature at this stage is still controllable until the generated combustible gases were gradually increased and then ignited which can be considered as the end of this stage.

The stage III began at Thermal runaway temperature (T_{TR}) which is marked by the temperature rising rate more than 10 degree-Celsius per second as shown in Figure 5.3 and then battery fire and explosion occurred in next few seconds. At this stage, the temperature of the battery cell cannot be controlled. The positive electrode and negative electrode were spread out from destroyed structure of battery cell as shown in Figure 5.4. However, gel-polymer based electrolyte and polymer-based separator were completely burnt out after the explosion process. Moreover, the voltage of battery cell became 0 V after explosion and no longer fluctuated which can be considered as the end of battery fire and explosion process.

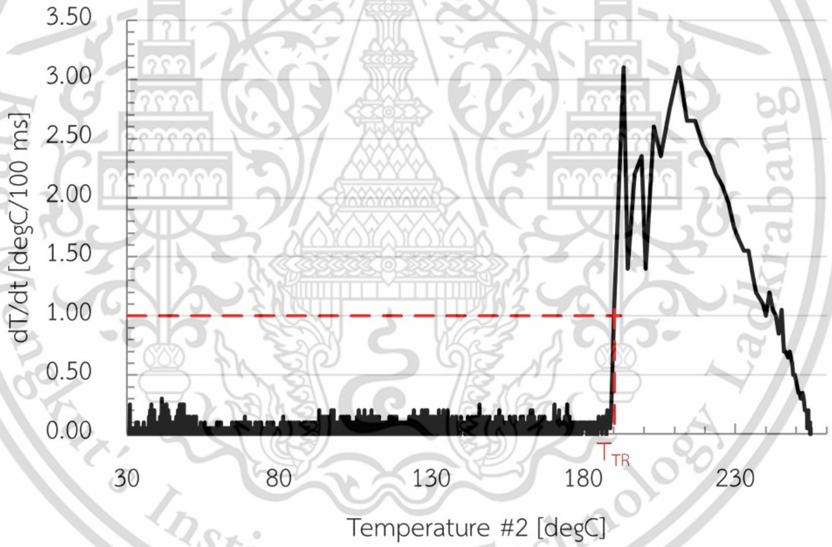


Figure 5.3 Surface temperature rise rate without liquid submersion suppression

Figure 5.4 Exploded cell after fire and explosion

5.2 THERMAL RUNAWAY PREVENTION AND FIRE SUPPRESSION

After obtaining the thermal runaway characteristic of TORIYAMA N18650CL-29 from previous experiment, the thermal runaway prevention and fire suppression using liquid-submerged technique was investigated. The liquid-submerged technique experiments to prevent thermal runaway and fire suppression were carried out by using three types of liquid fed into suppression chamber forcing liquid directly contact with battery cell to observe the possibility of each liquid suppression. For better understanding, the characteristic of voltage and temperature throughout using liquid-submerged technique is shown in Figure 5.5. The measured temperature and voltage were plotted on primary vertical axis and secondary vertical axis respectively. The result shows that the internal short circuit (ISC) occurred around 120 degree-Celsius (T_d) which is a bit different from previous experiment because of the difference of each cell quality. Furthermore, after using liquid-submerged technique to prevent thermal runaway, the voltage of cell suddenly drops from around 2.5 V down to nearly 0 V then fluctuates for a while and ended up with temperature cooling down to 40 degree-Celsius. Therefore, the voltage drop to 0 V indicates that using liquids not only cool down the temperature but also offer a safe method to stop hazardous from failure LIB. However, this still needs an additional explanation for possible reasons of the voltage drop mechanism which will be discussed in later pages.

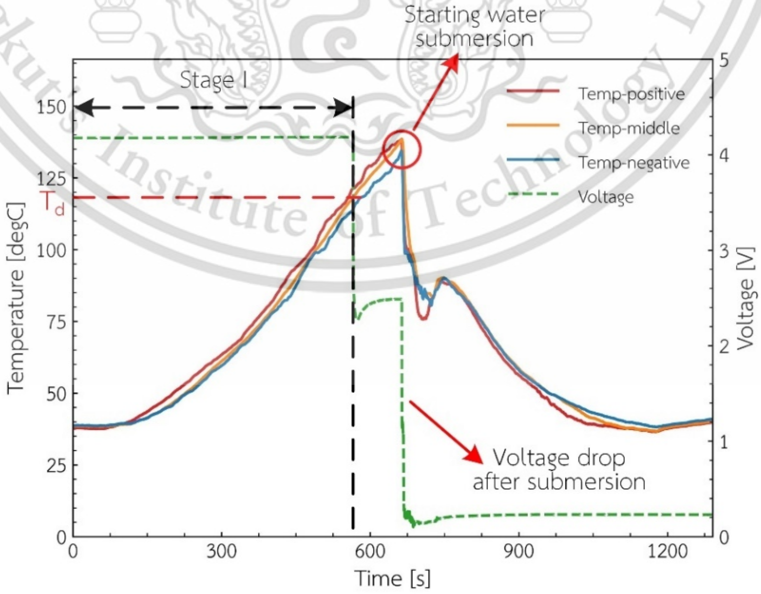


Figure 5.5 Voltage and surface temperature as a function of time entire liquid submersion experiment

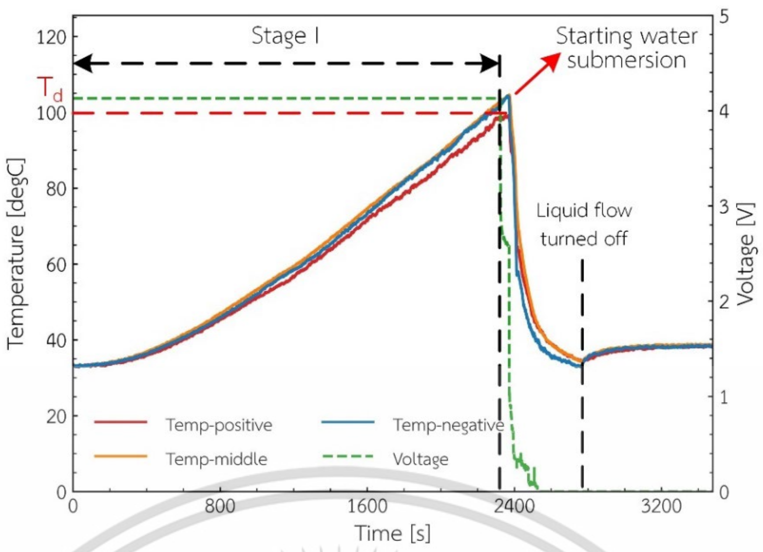


Figure 5.6 Voltage and surface temperature as a function of time entire DI water submersion

According to the difference of each cell's quality, it is impossible to use the equal value of voltage drop temperature (T_d) for triggering the liquid-submerged system. For that reason, the triggering device using voltage drop was used to build single cell liquid-submerged experiment for each liquid. In this experiment, after battery cell experienced overheated abuse condition, heater pad was turned off then each type of liquid will be fed into submersion chamber when voltage of battery cell begins to drop from fully charge 4.2 V to around 2.5 V. Video captures and images will be recorded to observed behavior of battery cell and liquid transformation due to chemical reactions during submersion process. Afterwards, dried out battery cells were observed terminal corrosion and deconstruction of battery structure to analyze the consequences hazardous from using liquid-submerged technique.

As shown in Figure 5.6, Figure 5.7 and Figure 5.8, measured temperatures as a function of time were plotted in red, orange, and blue solid line on primary vertical axis. On the other hand, measured voltage of cell was plotted in green-dash line on secondary vertical axis. The using of DI waster for liquid-submerged technique in Figure 5.6 implied that using liquid suppression for overheated abuse condition presents voltage drop down to 0 V right after suppression. The reason behind this phenomenon was probably thermal shock from cooling effect of using directly liquid submersion. When the aluminum tab as shown in Figure 2.3 undergoing sudden temperature changes, internal stress and strain will be developed inside material resulting in cracking of eventually failure. Due to the cracking and failure of aluminum tab, which is connected between cathode material, positive current collector and positive terminal of battery cell, the positive terminal of battery cell was separated from positive current collector inside the cell resulting in voltage drop to 0 V. Although it is a safe method for preventing thermal runaway and fire suppression, it still needs confirmation that the battery cell with fully energy stored inside but without any voltage cannot be dangerous.

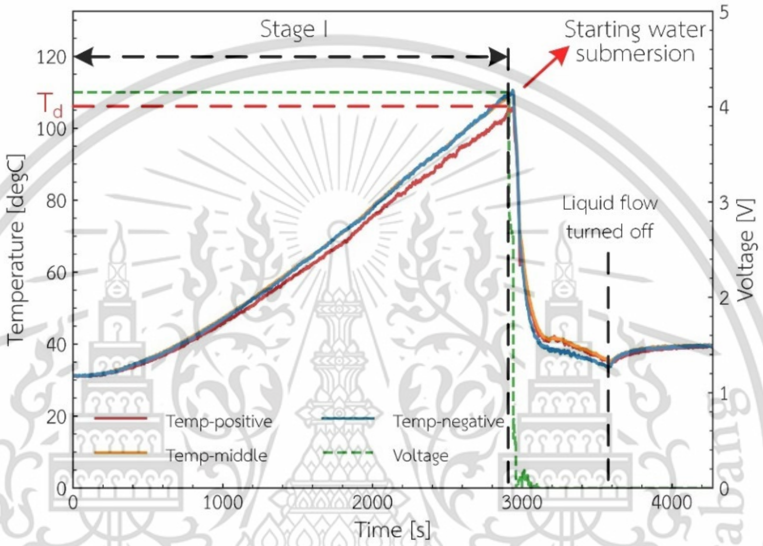


Figure 5.7 Voltage and surface temperature as a function of time entire SSW submersion

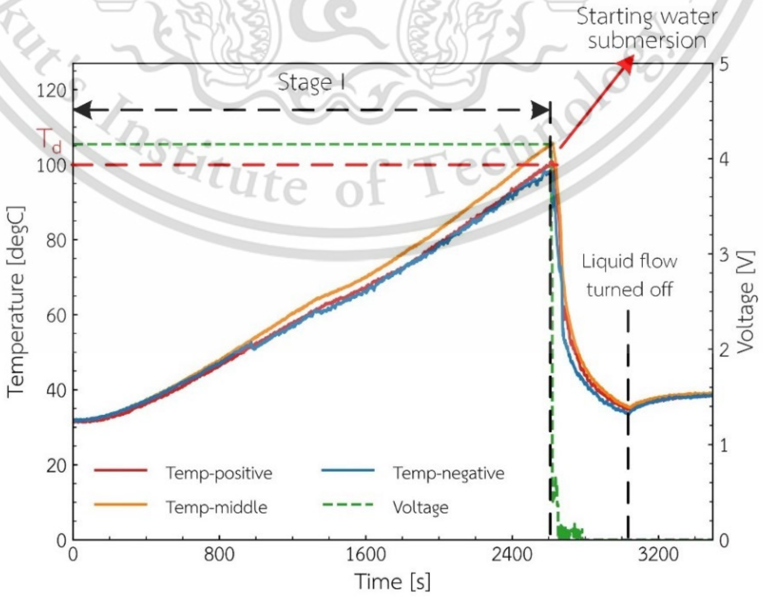


Figure 5.8 Voltage and surface temperature as a function of time entire Thai tap water submersion

Anyways, at the very first stage of investigation, this technique offers cooling effect then break the thermal runaway process which stops the consequences after battery cell experienced overheated abuse condition. However, after turned off liquid flowing system, the temperature slightly rose. This was not because the heat generated from battery cell due to thermal runaway, but because the heat from heater pad that still higher than battery cell and liquid temperature.

In addition, using SSW and Thai tap water as shown in Figure 5.7 and Figure 5.8 show the similar characteristic of using DI water. After starting water submersion when internal short circuit (ISC) occurs, the voltage of cell suddenly drops to 0 V. Nevertheless, it should be noted that the heat absorbed from liquids depended on the type and properties of each liquid, which is not considered in this research. From using three types of liquid submersion, all of liquid solutions offer voltage drop to 0 V confirming that using this technique preventing thermal runaway and fire suppression after the occurrence of internal short circuit (ISC) can be used effectively.

However, using voltage drop to trigger liquid suppression system is most suitable for only single cell, not in battery pack scale. The voltage drop occurred after internal short circuit (ISC) can be used as early warning of battery cell. On the other hand, only voltage drops in battery pack that consist of battery cell connected in series and parallel cannot be used to trigger suppression system due to its high voltage. The voltage drop only one cell in battery pack cannot caused significant drop for suppression system.

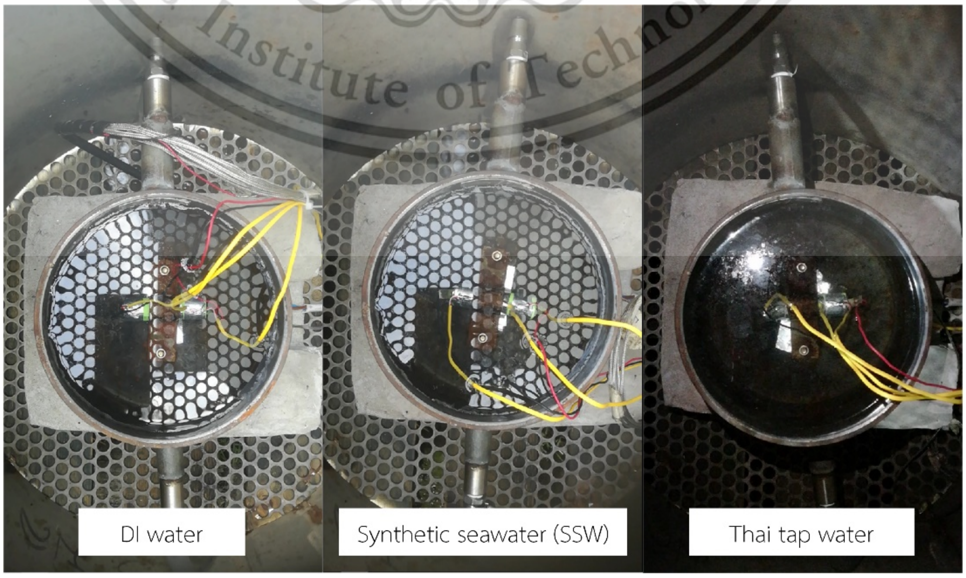


Figure 5.9 LIB and liquid solutions after submersion process

Because of voltage drop to 0 V, there was no longer voltage from battery cell to drives chemical reactions like water electrolysis reaction and the others causing terminal corrosion. Furthermore, without any terminal corrosion at positive terminal, the liquids used to submerge overheated cell still translucent for all three types of liquid which are DI water, SSW and Thai tap water as shown in Figure 5.9. As a result, using liquid submersion when battery cell is subjected to overheated abuse condition didn’t cause toxic solution like fully charged liquid submersion as shown I chapter 4. It is not dependent on the types of suppression liquid, but it is obviously depended on the thermal shock effect from this technique which means this technique can be used for any types of liquid.

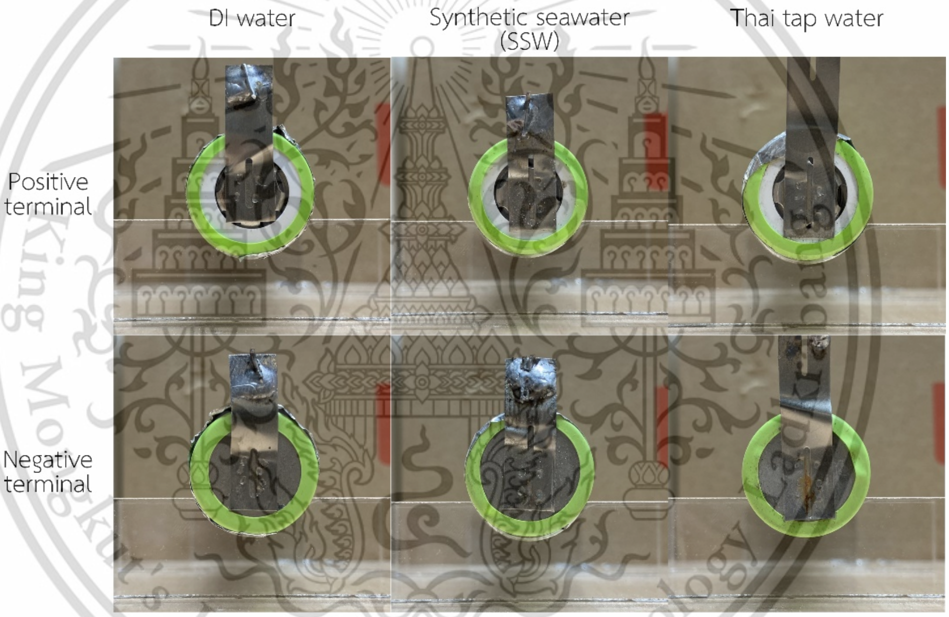


Figure 5.10 LIB terminal after liquids submersion experiment

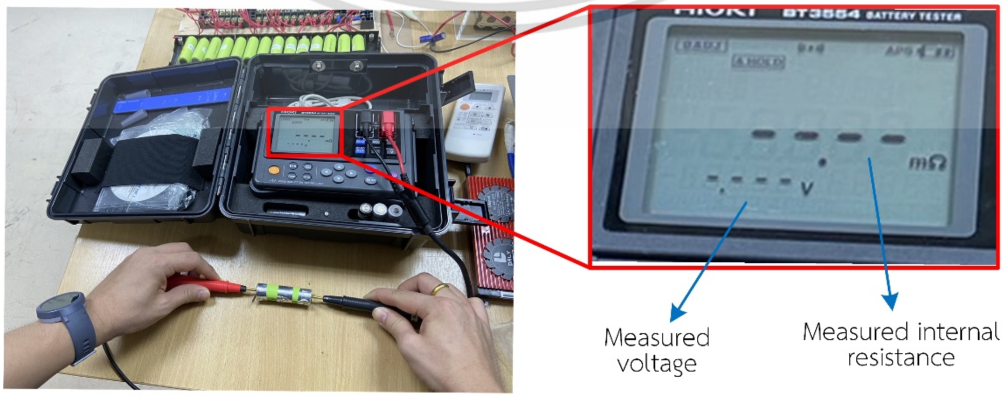


Figure 5.11 LIB Internal resistance measurement after liquids submersion

For terminal observation after thermal runaway prevention and fire suppression using liquid-submerged technique, the evidence shows that this technique can be used without any terminal corrosion and deposition at both positive and negative terminal as shown in Figure 5.10. The surfaces at positive and negative terminal of battery cell submerged in DI water were similar from pristine cell before using this technique while the terminal surfaces at positive and negative terminal of battery cell submerged in SSW had slightly salt, which are the composition of SSW, deposited on nickel tab at positive terminal and the surface of negative terminal. However, this deposition on surface of battery cell submerged in SSW was not a terminal corrosion and deposition as shown in Figure 4.9 because of the battery cell was submerged into SSW without any voltage driving chemical reactions. On the other hand, the negative terminal of battery cell submerged in Thai tap water shows little amount of rust due to the impurity of Thai tap water used in liquid submerged technique.

After thermal runaway prevention, all of battery cells were dried out for a week then using HIOKI BT3554 BATTERY TESTER to measure internal resistance. As shown in Figure 5.11, the voltage and internal resistance of battery cell cannot be measured after submersion. This scenario implied that using liquid-submerged technique can cause thermal shock at aluminum tab, which connected between positive current collector and positive terminal of battery cell, resulting in voltage and internal resistance cannot be measured.

CHAPTER 6

CONCLUSION AND RECOMENDATIONS

6.1 CONCLUSION

In this research, various types of liquids, which are DI water, synthetic seawater (SSW) and Thai tap water, were used to submerge cylindrical Lithium-ion battery with/without thermal abuse condition. Then, the consequence and the possibility of using liquid-submerged technique was investigated.

For various types of liquid submersion, when without thermal abuse condition, it was found that DI water submersion didn't cause any harm from terminal corrosion and toxicity after experiment. On the other hand, SSW submersion revealed that there was a liquid waste problem and severe terminal corrosion causing the occurrence of chemical reactions after using this technique but offered a potential to discharge voltage of submerged cell. Thai tap water submersion revealed that it is almost similar with DI water submersion, but the small amount of rust was observed on positive terminal. In addition, it was found that liquid circulated system increased the rate of discharged voltage, the corrosion reaction at positive terminal and chemical reaction occurred during SSW submersion.

From those results, it can be concluded that DI water and Thai tap water are an ideal solution and the most suitable for practical uses of liquid submersion technique respectively. Due to the toxicity after SSW submersion, using SSW submersion is considered to be used as an emergency liquid submersion. However, no fire and explosion observed throughout these experiments and all types of liquid used in this research had the potential to be used as submerged liquid for thermal runaway prevention and fire suppression system.

For the battery cell with overheated abuse condition, it was founded that the internal short circuit (ISC) occurred when surface temperature of battery cell reaches around 97.3 deg-Celsius causing voltage drop from 4.2 V to around 2.5 V. Afterwards, the safety vent valve began to vent with voltage fluctuations before explosion occurred. Then, all thermal runaway prevention and fire suppression experiments, which are DI water, SSW, and Thai tap water submersion, were performed by using voltage drop from 4.2 V to around 2.5 V to trigger liquid submersion system.

The experiments were investigated that using liquid-submerged technique after the voltage of battery cell dropped from 4.2 V to around 2.5 V caused the voltage of battery cell dropped to 0 V which is probably due to the cracking and the failure of aluminum tab at positive side for all types of liquid. In addition, due to the zero voltage of battery cell, there wasn't terminal corrosion and liquid waste problem after submersion.

This results implied that all types of liquid used in these experiments can be effectively used in Thermal runaway prevention and fire suppression system without any harm. SSW can be used as a reliable fire suppressant, especially when operating in the middle of the ocean. DI water shows great potential for use in both designed thermal runaway prevention systems and submerged cooling systems for electric vehicles. Moreover, tap water can be a suitable option for designed fire suppression systems by directly channeling it to the battery pack. However, the performance of cooling temperatures really depends on which specific type of liquid is used.

From those results, liquid-submerged technique, with all types of liquid, holds great potential for application in Thermal runaway prevention and fire suppression systems electric vehicles.

6.2 RECOMMENDATIONS

Because this work investigated only single cell of cylindrical Lithium-ion battery, further information of battery pack liquid submersion for thermal runaway prevention is needed. Moreover, the voltage drop triggering system in this work is not suitable for battery pack system because the voltage drop is hard to detect in higher voltage. Further triggering devices, such as pressure sensor, CO_2 sensor, temperature sensor etc., for thermal runaway prevention and fire suppression should be investigated which is our research topic for future work.

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APPENDIX A

PROCEEDING

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May 30, 2022

Certificate of Presentation

This is to certify that

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has participated in 2022 JSAE Annual Congress, held on May, 26, 2022,

at the Pacifico Yokohama, Yokohama, Japan(Hybrid event)

and orally presented the paper entitled: "Investigation of Cylindrical Lithium-Ion Battery Fire Suppression using Liquid-Submerged Technique ",

Presentation Number: 201. Paper Number: 20225201

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講演番号 201

文献番号 20225201

Investigation of Cylindrical Lithium-ion Battery Fire Suppression using Liquid-Submerged Technique

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ABSTRACT: Lithium ion battery (LIB) fires are dangerous and usually occur after a failure of battery. We demonstrates submersion of fully charged LIB in synthetic seawater, DI water and tap water, aiming to enlighten the knowledge on LIB fire suppression. The experiments were carried out by submersing full coverage of LIB cells. The voltage reduction was measured and thermal runaway (TR) was observed throughout the experiment. Then, the sediment in covered liquid were analyzed using XRD and FTIR to characterize products from experiment and organic electrolyte leakage. The results showed that Liquid-Submerged Technique has a possibility to suppresses fire from LIB.

KEY WORDS: **Safety, Suppression system** Submersion fire suppression, Battery submersion, Seawater submersion

1. INTRODUCTION

The lithium-ion battery (LIB) is commonly used as the main power source of electric vehicles (EVs) in recent years due to its high specific energy, light weight, long life cycles, low self-discharge rate, and low memory effect comparing to lead-acid battery and nickel-based battery [1–3]. However, the internal temperature of LIB becomes higher when operated on high charge/discharge rate, leading to thermal runaway (TR) which is a rapid and unstoppable increase of temperature in a sort of chain reaction and battery fire explosion [4–5].

There are electric boat fire accidents in Norway. In October 2019, the hybrid ferry "Ytteroyningen" caught fire and exploded 12 hours after fire extinguishing. Fires occurred when the ferry only ran on diesel engine then spread to battery compartment leading to battery thermal abuse condition [6–7]. Furthermore, there was a fire occurred near the battery compartment of a hybrid-electric boat in March 2021. After fire extinguishing, firefighters battled smoke and elevated temperature in battery for more than 24 hours to prevent fire re-ignition and explosion [8].

Comprehensive comparison indicates that water-based extinguishants are the most suitable and cost-effective for LIB fire extinguishing. It successfully suppresses LIB fire. Nevertheless, gases and smokes still occur after LIB fire extinguished and battery re-ignition might occurs after extinguished [4, 9–10]. Putting out the LIB fire is not only to suppress the fire from LIB but also require an effort to stop the chemical reactions inside LIB [2]. Water-based extinguishants can both extinguish the fire and cool down the burned surface, but it cannot inhibit LIB chemical reactions which is just prolongs burning process resulting in battery re-ignition. Therefore, the way to prevent battery re-ignition is by ensuring that LIB is completely burned out all the energy stored inside [9]. In case of electric ferry operated in the ocean, seawater, which acts as electrolyte and has capability to discharge battery, could be used as fire extinguishant to ensure that LIB is burned out. For this reason submersion battery into liquid and electrolyte solution is effective method to suppress LIB fire. It not only improves thermal performance but also, hinders the exothermic reaction and prevents reignition [11–15]. However, the liquid waste problem is still the main concern and need further research information to optimize conditions.

This research focused on submerging single cell to study possibility of LIB fire suppression using Synthetic seawater, DI water and tap water. Weinvestigated the LIB fire after liquid submersion, discharge rate, cell deconstruction and environmental pollution after process. The corrosion and cell deconstruction which lead to organic electrolyte leakage were analyzed. Moreover, safety submersion time which is the maximum time length to submerge LIB before serious corrosion occurs was elucidated to enlighten the knowledge on fire suppression from LIB using Liquid-submerged technique.

2. METHODOLOGY

2.1. Cylindrical Lithium-ion battery

The cell used in these experiments is commercial 18650 Lithium-ion cells with NMC ($LiNiMnCoO_2$) as a cathode of the cell. The typical structure of 18650 cell is shown in Fig. 1 [16]. The same type of TORIYAMA N18650CL-29 Lithium-ion cell was used throughout experiments as shown in Fig. 2 (a). All of the cells were charged following to standard charge method from previous report [17] using R&S®HMP4000 power supply and monitored temperature using FLIR E8 Thermal Imaging Camera throughout charging process as shown in Fig. 2 (b) and Fig. 2 (c). Moreover, cell weight was measured before experiments using METTLER TOLEDO Mettler Toledo ML-series. Cell specification is shown in Table 1.

Table 1 TORIYAMA N18650CL-29 Cell specification [17]

ITEM	SPECIFICATION
Nominal capacity [mAh]	2900 @0.2C
Nominal voltage [V]	3.6
Charge voltage [V]	4.2
Discharge cut-off voltage [V]	2.5
Cell dimension [mm]	Height: 64.85± 0.25 Diameter: 18.35± 0.15
Weight [g]	≤ 48
Standard charge method	CC-CV, Standardly 1375 mA, 4.2V (28 mA)

2.2. Synthetic seawater

Synthetic seawater was prepared by dissolving 35.675 grams of the main sea-salt in DI water with enough DI water added to make one-liter total solution. In this experiment, only 500 ml. synthetic seawater was used to submerge the cell in each experiment. The main sea-salt of synthetic seawater used in the experiment is shown in Table 2 [18].

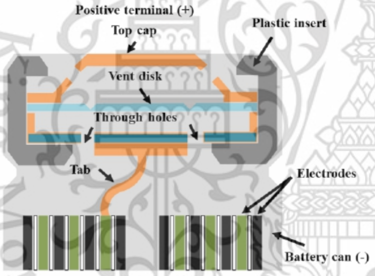

Fig. 1 The typical structure of 18650 battery cap [16]

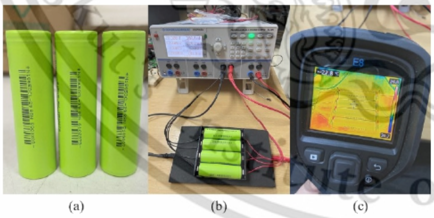

Fig. 2 (a) TORIYAMA N18650CL-29 cell used in experiment (b) charging process (c) thermal imaging camera

Table 2 Main chemical composition of synthetic seawater [18]

Compound	Concentration [g/L]
NaCl	24.53
$MgCl_2$	5.20
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.695
Total	**35.675**

2.3. Experimental procedures

After the cells were charged up to 4.2V (100% SOC) following to the standard charge method from Table 1, each cell was immersed into liquid batch, including Synthetic seawater batch, DI water batch and tap water batch collected from Pathum Thani province in Thailand, to start the liquid-submerged experiments. The sediment in covered liquid were characterized out by FTIR (Nicolet™ iS50R Research FTIR Spectrometer) and XRD (Rigaku - TTRAX III X-ray diffractometer) to investigate electrolyte organic leakage and products from submersion experiments respectively. Moreover, each cell was removed from the liquid 30 minutes intervals time to measure cell's voltage using handheld multimeter (HIOKI 3280-10F) and observe terminal corrosion physically then removed from liquid after 3 hours submersion as shown in Fig. 3.

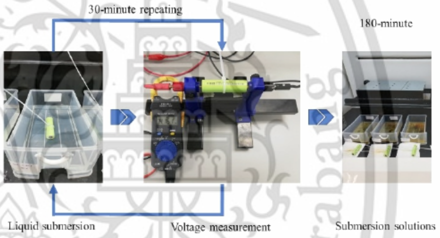

Fig. 3 Liquid-submerged experiments

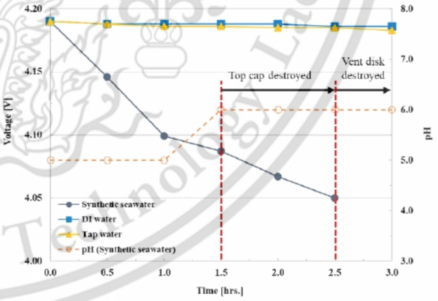

Fig. 4 Cell's voltage versus time curve

3. RESULTS AND DISCUSSIONS

3.1. Voltage and discharge rate

The cell voltage variation through immersion time is shown in Fig. 4. The results indicate slight reduction of battery voltage in DI water and tap water. On the other hand, synthetic seawater provided rapid discharge behavior to 4.05 V The cell's voltage could not be measured after 2.5 hours submersion time as shown in Fig. 4. Normally, submersion into electrolyte solution like seawater discharge energy inside LIB. However, the termination of voltage measurement in this case could be interpreted that vent disk or positive terminal was completely corroded, implying that battery was not fully discharged and still stored energy inside. However, there was no fire and explosion observed after submerging LIB into liquid in this experiments, which confirms that this liquid-submerged technique has potential to be used as LIB fire suppression.

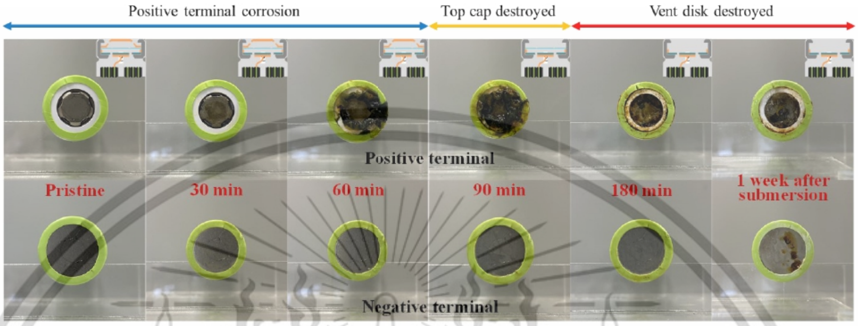

Fig. 5 Positive and negative terminal of the cell submerged in synthetic seawater

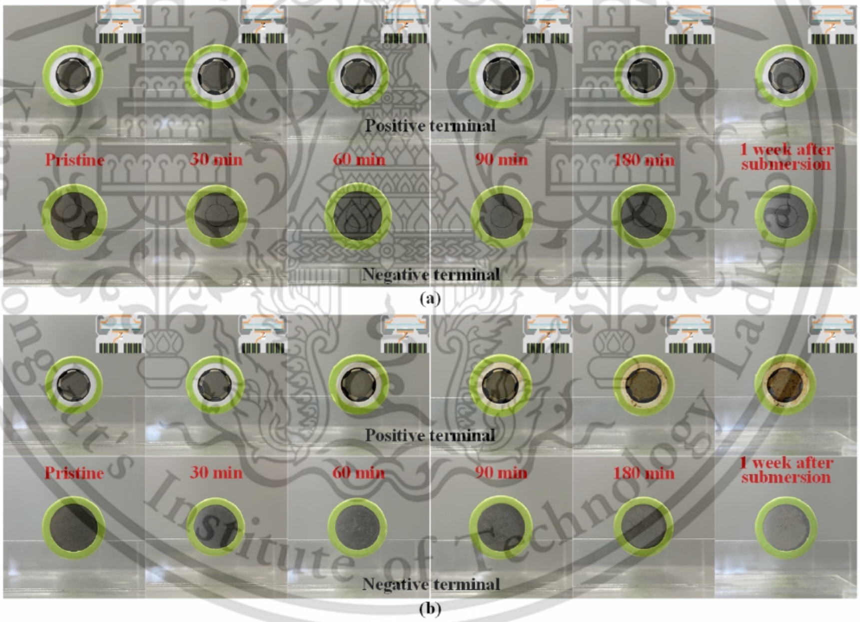

Fig. 6 Positive and negative terminal of the cell submerged in (a) DI water (b) Tap water

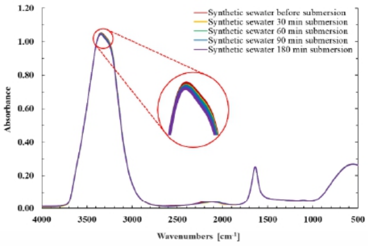

Fig. 7 FTIR result of sediments in synthetic seawater after liquid-submerged experiment

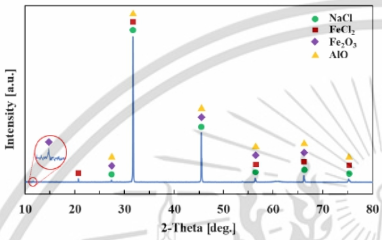

Fig. 8 XRD pattern of sediment in synthetic seawater after liquid-submerged experiment

3.2. Terminal corrosions

For synthetic seawater submersion, the top cap of positive terminal, mainly made of steel, was corroded after 1.5 hours submersion time as shown in Fig. 5. This was because Cl^- anions in synthetic seawater could facilitate metals dissolution resulting in serious galvanic corrosion on positive terminal [19] and the formation $FeCl_2$. Moreover, the cell exhibited black residue on positive terminal surface which is considered to be an iron oxide substances. After that, the vent disk of positive terminal was completely corroded in 2.5 hours submersion time, resulting in the failure of voltage measurement. Moreover, the organic electrolyte inside LIB was possibly leaked out and dissolved into liquid solution after this submersion time. This could be harmful to environment because Li salt in electrolyte might be react with water and form strong acid. On the negative terminal, the deposition of white substance was observed as shown in Fig. 5. This could be the salt composing of the cations in seawater solution (Na^+, Mg^{2+}, Ca^{2+}, K^+).

As shown in Fig. 6 (a) DI water seems like an ideal solution for this technique because the serious corrosion occurred on positive terminal of the cell was not observed. For tap water in Fig. 6 (b), there was small amount of rust on positive terminal and also small amount of metal deposited on negative surface due to less some metal impurity and chloride composition in liquid. However, it can be considered as minor terminal corrosion without any harms and toxicity from the leakage of organic substances inside LIB. Therefore, it can be used as practical LIB fire suppressant without fire and explosion.

3.3. Weight loss

Table 3 shows the cell weight measured before and after liquid-submerged experiment. The cell in synthetic seawater has lost weight around 1.6 grams due to its serious corrosion on positive terminal. However, a small weight difference can be observed for the cell in DI water and tap water.

Table 3 cell's weight before and after experiment

Liquids	Before	After	Weight change [g]
	Weight [g]		
Synthetic seawater	46.5679	44.9853	-1.5826
DI water	46.8384	46.8353	-0.0031
Tap water	46.6460	46.6483	0.0023

3.4. Solution characterization

After 1 week later of experiment, there was organic electrolyte leaked out from LIB at positive terminal of the cell submerged in seawater as shown in Fig. 5. The leaked substances looked like a gel which can confirm that this type of LIB used gel polymer electrolyte instead of liquid electrolyte. However, there was a risk that a gel polymer electrolyte dissolved into solution. The FTIR characterization was used to identify dissolved organic substances in synthetic seawater after submersion experiment.

The submerged synthetic seawater collected from liquid-submerged experiment was dried overnight at 80 degree-Celcius and the sediment was collected and characterized. The characterization result is shown in Fig. 7. It could be seen that there was no difference in any FTIR spectra, implying that there was no organic substances detected in any of collected solutions. This means that submerging cell into seawater for 2.5 hours didn't make the gel organic electrolyte dissolved into submerged solution. However, during the submersion experiment, the solution turned to yellow and then orange/brown indicating that positive terminal corrosion generated iron rust and the oxygen compounds of iron including $FeCl_2$ and Fe_2O_3 as shown in XRD pattern in Fig. 8. Moreover, after 1.5 hours submersion time, the generated white substance was later proved as an aluminum oxide (AlO) in Fig. 8.

As a result, the generated oxygen compounds of iron and Aluminum oxide from main composition of cell's positive terminal before and after 1.5 submersion time proved that using synthetic seawater as a fire suppressant makes liquid waste problem toward the environment. Moreover, 1.5 hours or 90 minutes submersion time for synthetic seawater can be considered as the safety submersion time before serious corrosion destroyed positive terminal completely increasing the risk of organic electrolyte leakage.

4. CONCLUSION

The LIB cells were immersed into Synthetic seawater, DI water and tap water to study possibility of LIB fire suppression. The positive terminal of cell submerged in Synthetic seawater was completely corroded after 2.5 hours submersion time. The top cap of positive terminal was destroyed after 1.5 hours submersion time, which can be considered as safety submersion time. However, synthetic seawater submersion still end up with liquid waste problems because it contains product from electrolysis reaction in submerged liquid. Moreover, submersion in synthetic seawater can provide the voltage discharging to reduce the risk of LIB re-ignition. Nevertheless, using seawater as fire suppressant is acceptable in emergency case but the handling of liquid waste after suppression needs to be further considered.

Furthermore, DI water is an ideal liquid for LIB fire suppression because of no terminal corrosion occurred. Tap water is most suitable in practical use for LIB fire suppression because of its low impact on terminal corrosion. However, both DI water and tap water doesn't provide the voltage discharged to reduce the risk of LIB re-ignition.

ACKNOWLEDGEMENTS

This study is supported by researchers, facilities and resources for experiments from National Energy Technology Center (ENTEC), and from King Monkut's Institute of Technology Ladkrabang (KMITL). Most thankful to Thailand Advanced Institute of Science and Technology (TAIST) for financial support.

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