CHAPTER 4

RESULTS AND DISCUSSION

The experimental results and discussion was reported in this chapter that can be divided to three sections. Part1 synthesized results of Al-Zn alloy cathode and Cu anode. Part 2 characteristic composed of crystal structure, microstructure, density, hardness and resistance) of Al-Zn alloy cathode and Cu anode. Part 3 showed the results of electrochemical properties. electrochemical cells fabrication and electrochemical batteries application.

SYNTHESIZED Al-Zn ALLOY CATODE AND Cu ANODE

Figure 46 Al-Zn alloy cathode and Cu anode

Figure 46 showed the sample of Al-Zn alloys cathode and Cu anode by hotpress method. Disc samples which used in this study for 2.0 cm diameter, 1 cm thickness of Al, Al-Zn alloys cathode and the Cu anode of 0.1 cm.

CRYSTAL STRUCTURE OF Al-Zn alloy CATHODE AND Cu ANODE
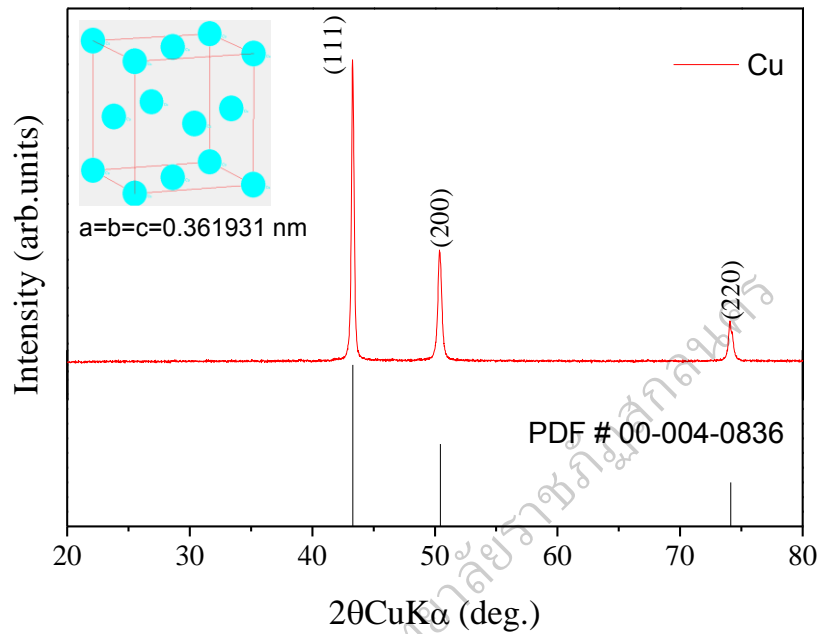
1. Crystal Structure of Cu

Figure 47 X-ray diffraction patterns of Cu anode

Figure 47 the crystal structure of Cu anode was obtained at 900 ^{o}C pressed 1.0 MPa found that Cu was cubic structure correspond with PDF#00-004-0836, lattice parameters were calculated using equation number 3.2 that is a = b = c = 3.6193±0.0657 Å.
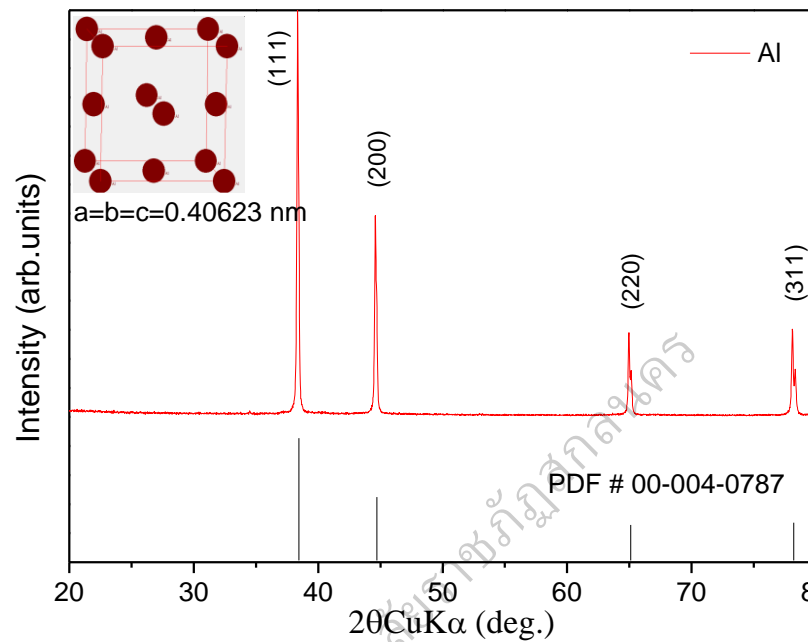
2. Crystal Structure of Al

Figure 48 X-ray diffraction patterns of Al cathode

Figure 48 the crystal structure of Al cathode was obtained at 670 ^{o}C pressed 1.0 MPa found that Al was cubic structure correspond with PDF#00-004-0787, lattice parameters were calculated using equation number 3.2 that is a = b = c = 4.0623±0.0072 Å.

Figure 47 and 48 were confirmed single phase of Cu anode and Al cathode indicate that the successfully preparation of high purity of metal by hotpress method.
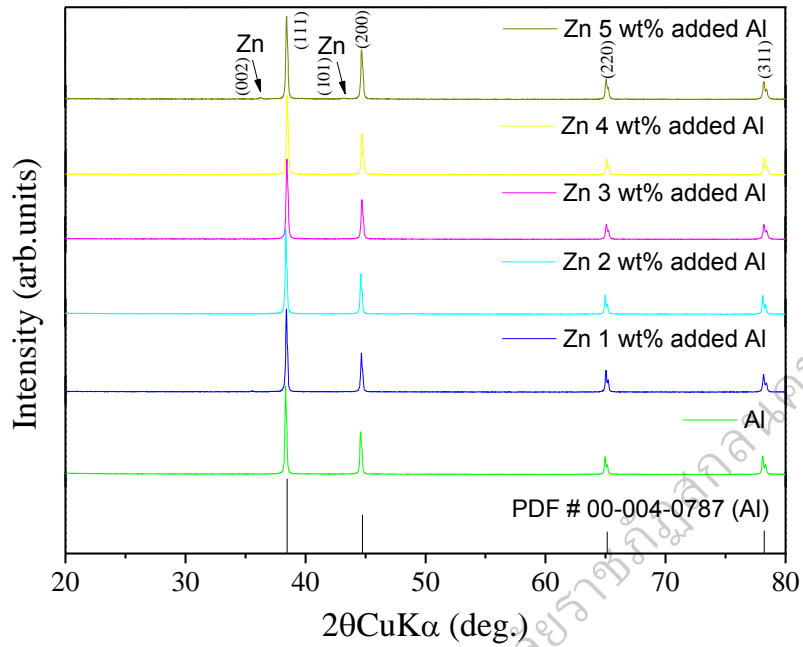
3. Crystal Structure of Alloy Al-Zn

Figure 49 X-ray diffraction patterns of Al and alloy Al-Zn (1-5 wt%)

Figure 49 showed the crystal structure Al doped Zn (0, 1, 2, 3, 4 and 5 wt%) confirmed that Zn phase appeared at 5 wt% doped Al cathode. It indicated that successfully prepared by hotpress method.

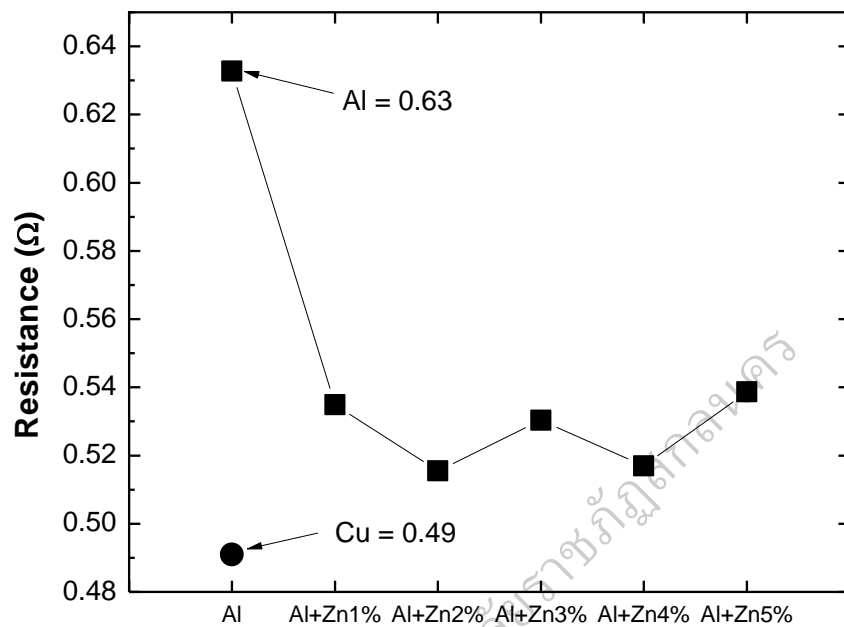# RESISTANCE OF Al-Zn ALLOY CATHODE AND Cu ANODE

Figure 50 The resistance of pure Cu, Al and alloy of Al-Zn (1-5 by wt%)

Figure 50 showed the resistance of samples the resistance of Cu anode Al doped Zn (1, 2, 3, 4 and 5 wt %). It found that the resistance of Al decreased with increasing Zn contents. It noted that Zn atoms substituted into vacancy atoms.

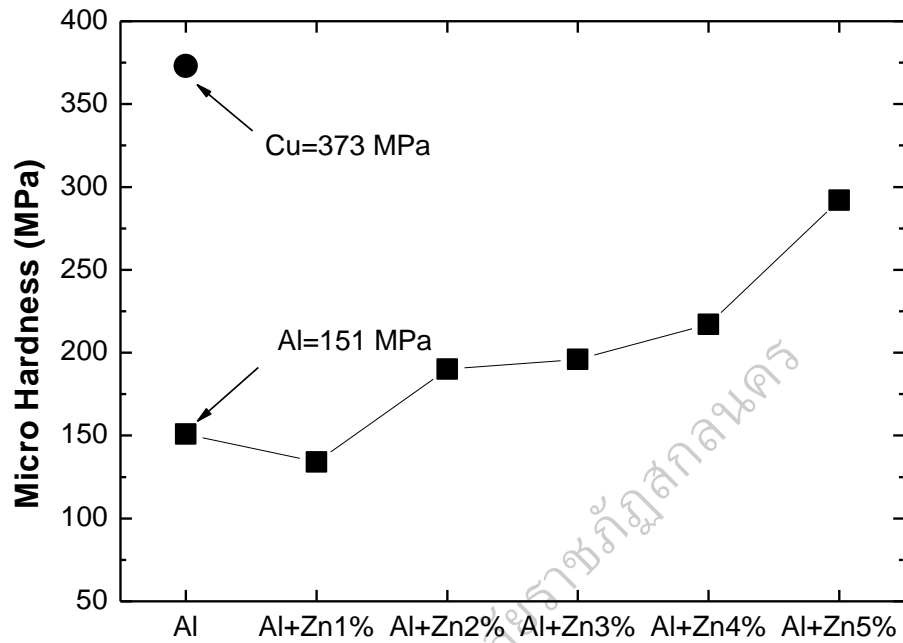## VICKER MICROHARDNESS OF Al-Zn ALLOY CATHODE AND Cu ANODE

Figure 51 The Vicker microhardness of Cu, Al and alloy of Al-Zn (1-5 wt%)

Figure 51 showed the Vicker microhardness of samples the Vicker hardness of Cu anode Al doped Zn (1, 2, 3, 4 and 5 wt %). It found that the Vicker hardness of Al increased with increasing Zn contents due to temperature in hotpress process which nearly melting point of Al.

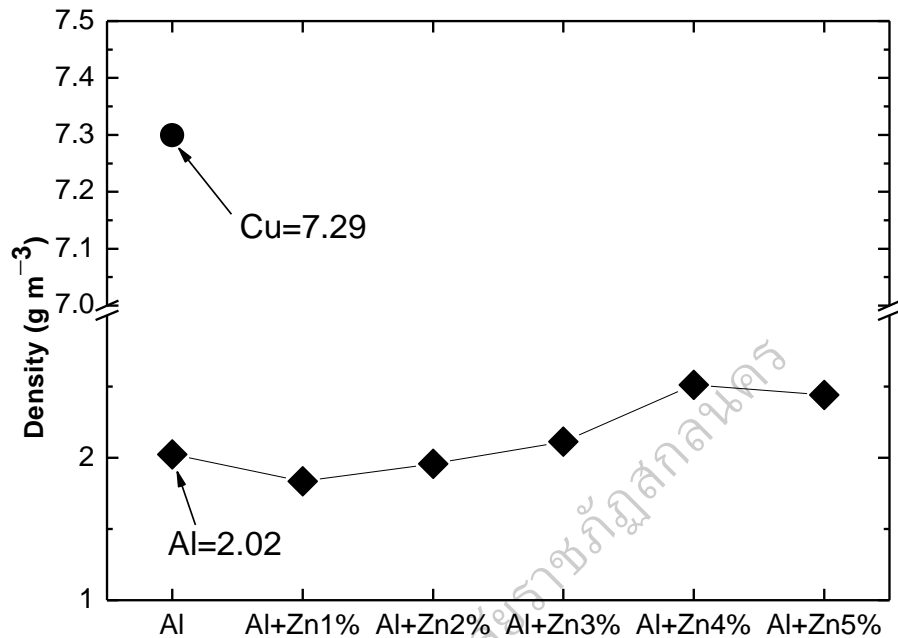## DENSITY OF Al-Zn ALLOY CATHODE AND Cu ANODE

Figure 52 The density of pure Cu, Al and alloy of Al-Zn (1-5 wt%)

Figure 52 showed the density of samples the density of Cu anode Al doped Zn (1, 2, 3, 4 and 5 wt %). It found that the density of Al increased with increasing Zn contents due to temperature in hotpress process which nearly melting point of Al.

MICROSTRUCTURE OF Al-Zn ALLOY CATHODE AND Cu ANODE

The microstructure of before and after dip in NaCl (2 mol dm^{-3}) + DI water (100 ml) for 40 min and then annealing at 60 $^{\circ}$C for 16 h were observed, are show in Figure 53 and 54.
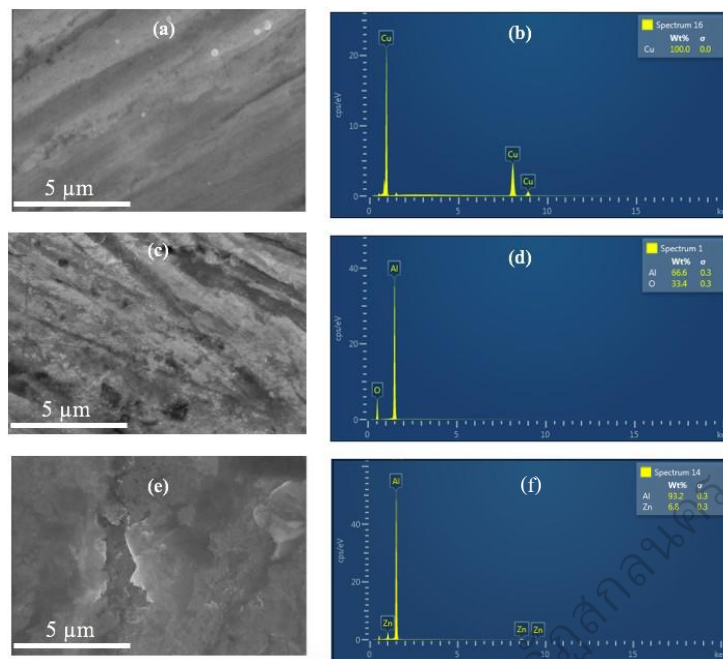
Figure 53 The SEM images of (a) pure Cu (c) Al (e) Al-Zn3 wt%, EDS images of
(b) Cu (d) Al (f) Al-Zn3 wt% before dip in NaCl+ DI water solution

Figure 53 showed the microstructure surface and elemental
compositions of Cu, Al, and Al-Zn3wt% by scanning electron microscopy (SEM)
and energy dispersive X-ray spectrophotometry (EDS) respectively. It found that
Cu surface such a smooth more than Al surface as shown Figure 53 (a) and (c),
suggested that was strength bond of Cu atoms. Elemental compositions of Cu
and Al as shown in Figure 53 (b) and (d) which confirmed that metal powder of
Cu and Al. In this study found that Al-Zn3wt% showed median values of
samples, then Al-Zn3wt% surface and element compositions was presented as in
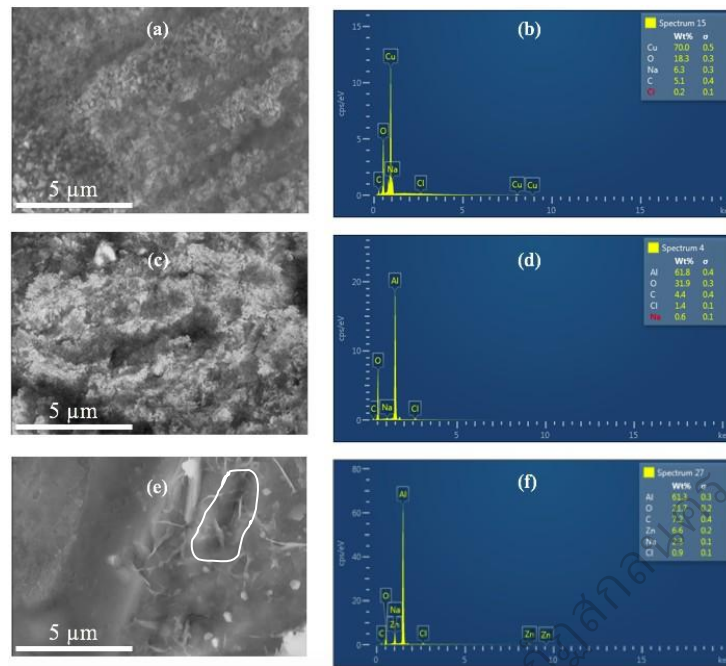Figure 53 (e) and (f).

Figure 54 The SEM images of (a) pure Cu (c) Al (e) Al-Zn3wt%, EDS images of
(b) Cu (d) Al (f) Al-Zn3wt% after dip in NaCl+ DI water solution

Figure 54 showed the microstructure surface and elemental compositions of Cu, Al, and Al-Zn3wt% after dipping in NaCl (2 mol dm^{-3}) + DI water (100 ml) for 40 min and then annealing at 60 $^{\circ}$C for 16 h by scanning electron microscopy (SEM)and energy dispersive X-ray spectrophotometry (EDS) respectively. It found that Cu was rough surface less than Al surface as shown Figure 54 (a) and (c), suggested that was strength bond of Cu atoms. Elemental compositions of Cu and Al as shown in Figure 54 (b) and (d) which seen that Na and Cl attached both Cu and Al surface. In this study found that Al-Zn3wt% surface showed larger hole than before dipping Al-Zn3wt% into solution seen in Figure 54 (e). Meanwhile elemental compositions for wt% of Al and Zn in Al-Zn3 wt% has deceased as followed in Figure 54 (f). It noted that ionization process of Al and Zn in NaCl solution.

ELECTRICAL CHEMISTRY PROPERTIES OF An-Zn ALLOY CATHODE AND Cu ANODE
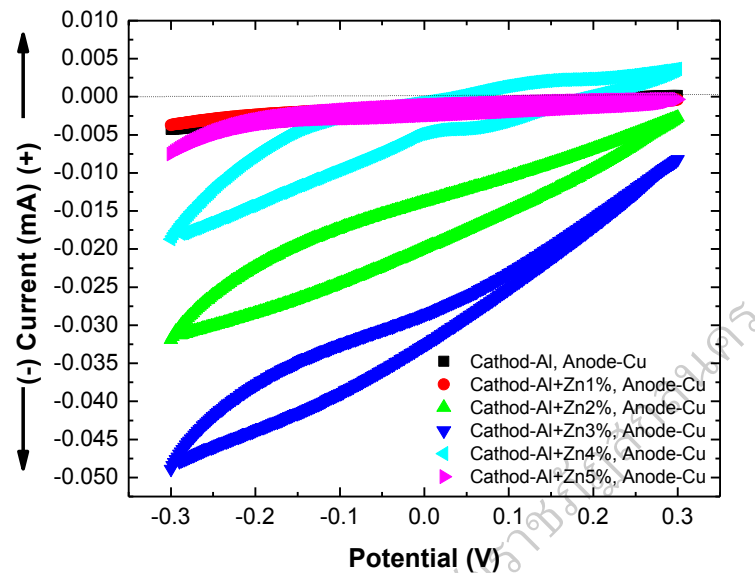

Figure 55 Voltammetric behavior of Cathode Al-Zn (1-5 wt%). and anode Cu for 100 Cyclic Voltammetry technique

Figure 55 showed current as a function of potential of Cu, Al and Al-Zn (1-5 wt%). It found that Al-Zn3wt% has optimized for current and potential, it mean that Al-Zn3wt% appropriate to apply for cathode and anode due to high current and low potential.

BALANCE MASS OF An-Zn ALLORY CATHODE AND Cu ANODE

Table 6 Mass of pure Cu, Al and alloy of Al-Zn (1-5 wt%) before and
after dip in NaCl+ DI water solution

Sample	Before(g) dip	After(g) dip	difference (g)
Cu1	2.6019	2.6105	0.0086
Cu2	2.4198	2.4295	0.0097
Cu3	2.7138	2.7226	0.0088
Cu4	2.7483	2.7634	0.0151
Cu5	2.4277	2.4388	0.0111
Cu6	3.2677	3.2791	0.0114
Al	1.2042	1.1636	-0.0406
AlZn1	1.2112	1.1876	-0.0236
AlZn2	1.2913	1.2661	-0.0252
AlZn3	1.4092	1.3996	-0.0096
AlZn4	1.5211	1.5	-0.0211
AlZn5	1.6411	1.639	-0.0021

Table 6 showed mass balance of Cu, Al and alloy of Al+Zn (1-5 wt%)
before and after dip in NaCl+ DI water solution for 40 min. It found that
maximum weight loss for Al, minimum weight loss for Al-Zn5wt%, which Zn atom
could decreased corrosion NaCl solution, corresponding Vicker microhardness
and density results.

DISCHARGING OF Al-Zn ALLORY CATHODE AND Cu ANODE
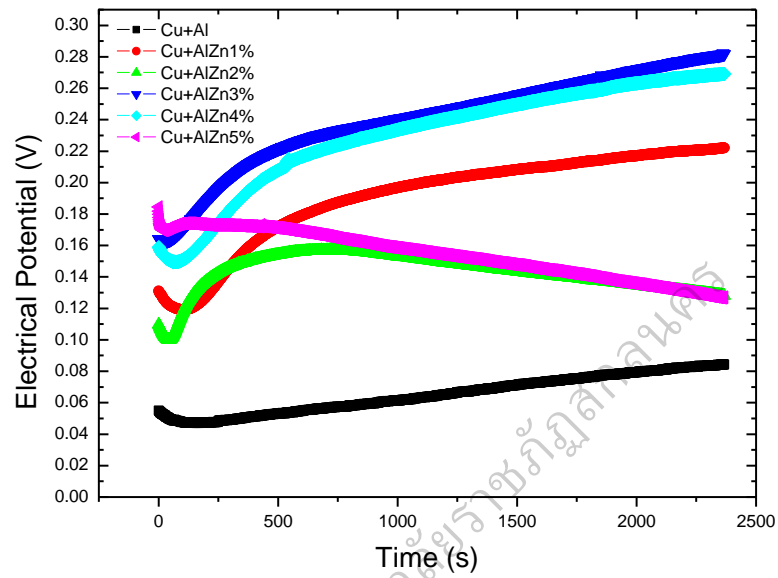
1. Electrical Potential

Figure 56 Relationship time with electric potential after dip Cathode Al, Al-Zn
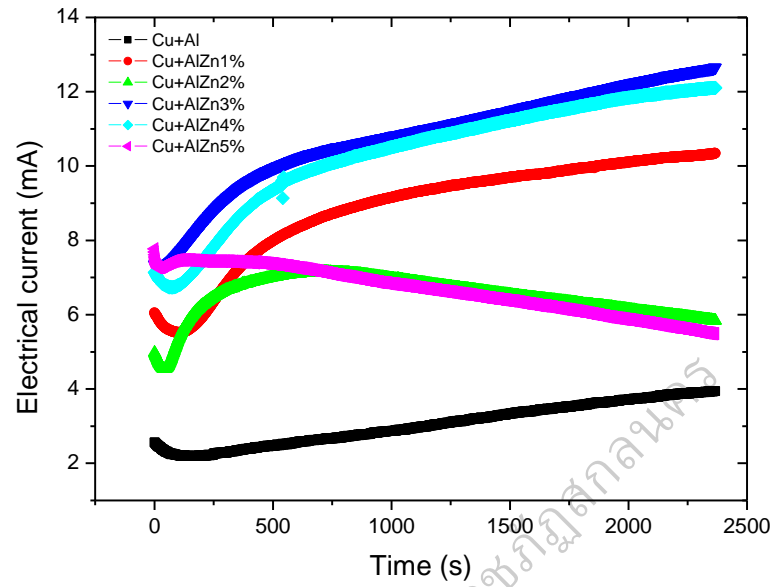and anode Cu in aqueous NaCl

2. Electrical Current

Figure 57 Relationship time with electric current after dip Cathode Al, Al-Zn

And anode Cu in aqueous NaCl
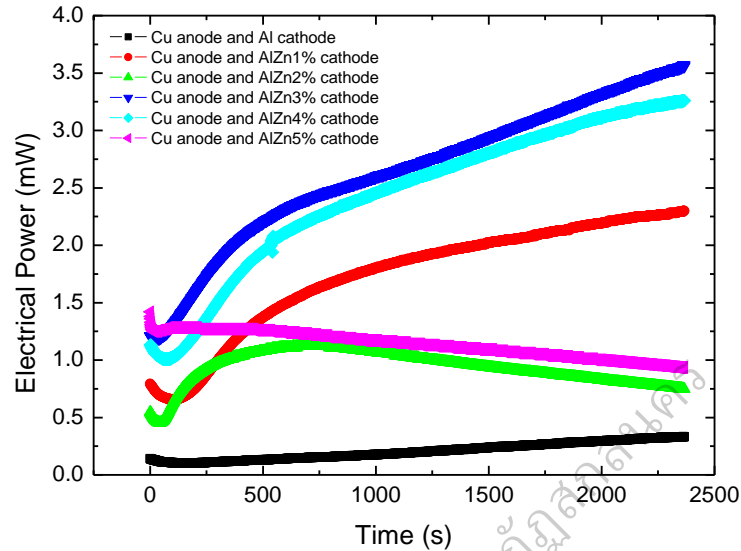
3. Electrical Power

Figure 58 Relationship time with electric power after dipping cathode Al-Zn
and anode Cu in aqueous NaCl

Figure 55, 56 and 57 electrical potential, electrical current and
electrical power as a function of time respectably. It found that Al-Zn3wt%
showed maximum of potential, current and power because it has homogenized
for adding Zn atom into Al structure of 3wt% which corresponding to cyclic
voltemmetry.

ELECTROCHEMICAL BATTERIES FABRICATION

Electrochemical batteries fabrication module 4 cells of Al-Zn alloy
cathode best condition and Cu anode and applied turn on a LED lamp.

Figure 59 Electrochemical battery fabricated 4 cells

Figure 60 Electrochemical battery fabricated 4 cells and applied

turn on a LED lamp.

Figure 59 and 60 electrochemical batteries fabrication module 4 cells of Al-Zn3wt% alloy cathode were chosen for applied with Cu anode and LED lamp used for load.
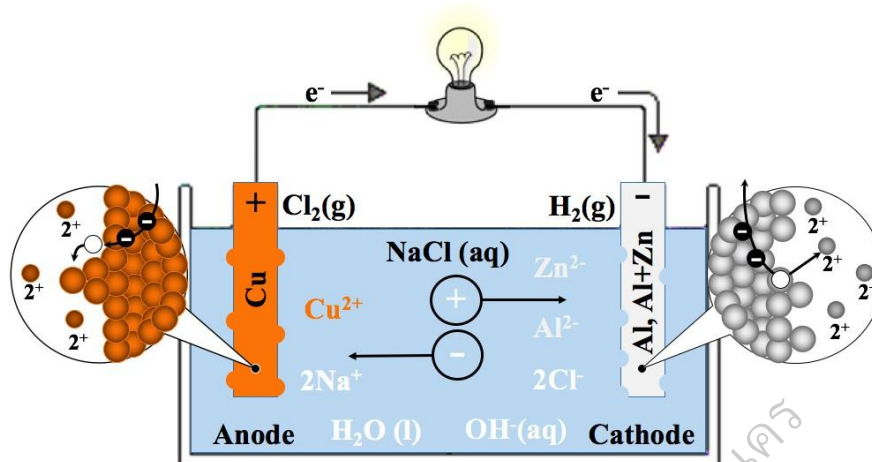
DISCHARGING

Figure 61 discharging of electrochemical cell

An apparatus that is used to generate electricity from a spontaneous redox reaction or, conversely, that uses electricity to drive a nonspontaneous redox reaction is called an electrochemical cell.

There are two types of electrochemical cells: galvanic cells and electrolytic cells. A galvanic (voltaic) cell Galvanic cells are named for the Italian physicist and physician Luigi Galvani (1737–1798), who observed that dissected frog leg muscles twitched when a small electric shock was applied, demonstrating the electrical nature of nerve impulses uses the energy released during a spontaneous redox reaction (ΔG < 0) to generate electricity. This type of electrochemical cell is often called a voltaic cell after its inventor, the Italian physicist Alessandro Volta (1745–1827). In contrast, an electrolytic cell consumes electrical energy from an external source, using it to cause a nonspontaneous redox reaction to occur (ΔG > 0). Both types contain two electrodes, which are solid metals connected to an external circuit that provides an electrical connection between the two parts of the system. The oxidation half-reaction occurs at one electrode (the anode), and the reduction half-reaction occurs at the other (the cathode). When the circuit is closed, electrons flow from the anode to the cathode. The electrodes are also connected by an electrolyte, an

ionic substance or solution that allows ions to transfer between the electrode compartments, there by maintaining the system's electrical neutrality. In this section, we focus on reactions that occur in galvanic cells.

A galvanic cell (left) transforms the energy released by a spontaneous redox reaction into electrical energy that can be used to perform work. The oxidative and reductive half-reactions usually occur in separate compartments that are connected by an external electrical circuit; in addition, a second connection that allows ions to flow between the compartments (shown here as a vertical dashed line to represent a porous barrier) is necessary to maintain electrical neutrality. The potential difference between the electrodes (voltage) causes electrons to flow from the reductant to the oxidant through the external circuit, generating an electric current. In an electrolytic cell (right), an external source of electrical energy is used to generate a potential difference between the electrodes that forces electrons to flow, driving a nonspontaneous redox reaction; only a single compartment is employed in most applications. In both kinds of electrochemical cells, the anode is the electrode at which the oxidation half-reaction occurs, and the cathode is the electrode at which the reduction half-reaction occurs.

The figure below shows an idealized drawing of a cell in which an aqueous solution of sodium chloride is electrolyzed.

Once again, the Na+ ions migrate toward the negative electrode and the Cl- ions migrate toward the positive electrode. But, now there are two substances that can be reduced at the cathode: Na+ ions and water molecules.

Cathode (-):

$$Na^+ + e^- \longrightarrow Na \qquad\qquad E_{ored} = -2.71 \text{ V} \qquad\qquad (4.1)$$

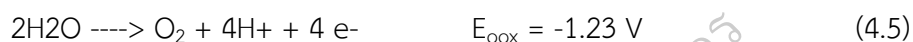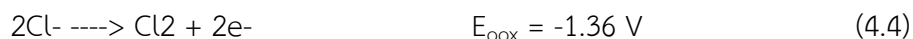$$2H_2O + 2e^- \longrightarrow H_2 + 2OH^- \qquad\qquad E_{ored} = -0.83 \text{ V} \qquad\qquad (4.2)$$

Because it is much easier to reduce water than Na+ ions, the only product formed at the cathode is hydrogen gas.

Cathode (-):

$$2H_2O \text{ (l)} + 2 \text{ e-} \longrightarrow H_2 \text{ (g)} + 2OH^- \text{ (aq)} \qquad (4.3)$$
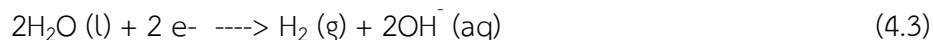
There are also two substances that can be oxidized at the anode: Cl^- ions and water molecules.

Anode (+):

$$2Cl^- \longrightarrow Cl2 + 2e^- \qquad E_{oox} = -1.36 \text{ V} \qquad (4.4)$$

$$2H2O \longrightarrow O_2 + 4H^+ + 4 \text{ e-} \qquad E_{oox} = -1.23 \text{ V} \qquad (4.5)$$

The standard-state potentials for these half-reactions are so close to each other that we might expect to see a mixture of Cl_2 and O_2 gas collect at the anode. In practice, the only product of this reaction is Cl_2.

Anode (+):

$$2Cl^- \longrightarrow Cl_2 + 2e^- \qquad (4.6)$$
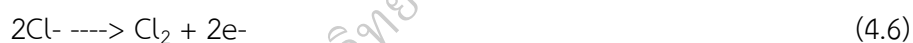
At first glance, it would seem easier to oxidize water (E_{oox} = -1.23 volts) than Cl- ions (E_{oox} = –1.36 volts). It is worth noting, however, that the cell is never allowed to reach standard-state conditions. The solution is typically 25% NaCl by mass, which significantly decreases the potential required to oxidize the Cl^- ion. The pH of DI water of 8.0 and water + NaCl of 7.3 the cell is also kept very high, which decreases the oxidation potential for water. The deciding factor is a phenomenon known as overvoltage, which is the extra voltage that must be applied to a reaction to get it to occur at the rate at which it would occur in an ideal system. Under ideal conditions, a potential of 1.23 volts is large enough to oxidize water to O_2 gas. Under real conditions, however, it can take a much larger voltage to initiate this reaction. (The overvoltage for the oxidation of water can be as large as 1 volt.) By carefully choosing the electrode to maximize the overvoltage for the oxidation of water and then carefully controlling the

potential at which the cell operates, we can ensure that only chlorine is produced in this reaction.
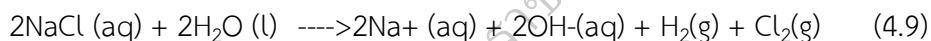
In summary, electrolysis of aqueous solutions of sodium chloride doesn't give the same products as electrolysis of molten sodium chloride. Electrolysis of molten NaCl decomposes this compound into its elements.

electrolysis

$$2NaCl(l) \quad ----> \quad 2Na(l) + Cl_2(g) \tag{4.8}$$

Electrolysis of aqueous NaCl solutions gives a mixture of hydrogen and chlorine gas and an aqueous sodium hydroxide solution.

electrolysis

$$2NaCl\ (aq) + 2H_2O\ (l) \quad ---->2Na+\ (aq) + 2OH\text{-}(aq) + H_2(g) + Cl_2(g) \tag{4.9}$$

Because the demand for chlorine is much larger than the demand for sodium, electrolysis of aqueous sodium chloride is a more important process commercially. Electrolysis of an aqueous NaCl solution has two other advantages. It produces H_2 gas at the cathode, which can be collected and sold. It also produces NaOH, which can be drained from the bottom of the electrolytic cell and sold.

The dotted vertical line in the above figure represents a diaphragm that prevents the Cl_2 produced at the anode in this cell from coming into contact with the NaOH that accumulates at the cathode. When this diaphragm is removed from the cell, the products of the electrolysis of aqueous sodium chloride react to form sodium hypo-chlorite, which is the first step in the preparation of hypochlorite bleaches, such as Chlorox.