School of Chemical Sciences
UNIVERSITI SAINS MALAYSIA

ELECTROLYSIS OF AQUEOUS COPPER (II) SULPHATE AND SODIUM HYDROXIDE & ELECTROCHEMISTRY: ELECTROPLATING HANDBOOK

http://chem.usm.my

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LIST OF AWARDS

RECEIVED GOLD AWARDS FOR:

i-IDeA™ 2018
THE 4TH INTERNATIONAL INNOVATION, DESIGN AND ARTICULATION

UNIVERSITI TEKNOLOGI MARA
PERLIS BRANCH

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INTRODUCTION

This handbook is prepared in order to integrate the microscale approach into the chemistry curriculum of Malaysian secondary schools taking into consideration the limited funding in terms of chemicals, glassware and laboratory facilities.

The programme aims at reaching out to students as well as teachers to become involved in chemistry as a subject in order to increase their interest in science besides promoting science in a smaller scale. In our part, miniaturized or microscale chemistry is part of our attempts to solve problems related to limited chemicals. Besides attending lectures on selected topics, teachers will also be involved in conducting new experiments using USM new glass-based miniaturized microscale kits developed by our staff.

COMPONENTS IN THE KIT

Components	Quantity
Combo plate (24 well)	1
Cloth peg	1
Copper foil	1
Eelctrolysis set	1
Iron nail	2
Led set	1
Propette	2
Test tube 5 mL	2

COMPONENTS IN THE KIT

Chemicals	Volume (mL)
1 mol dm^{-3} $CuSO_4$	5
1 mol dm^{-3} $CuSO_4$	10
1 mol dm^{-3} NaOH	10

Exp 1: Electrolysis of Aqueous Copper (II) Sulphate and Sodium Hydroxide

Objective:

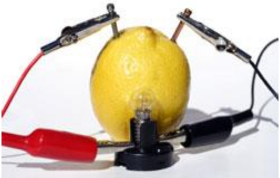
To investigate the electrolysis of aqueous copper (II) sulphate, $CuSO_4$ solution and sodium hydroxide, NaOH solution.

Engage:

The teacher shows a rusted nail and asks the following questions:

- What are the causes of rusting?

The teacher conducts a demonstration using a good nail and rusty nail (see picture).



The teacher will create some dialogues with the students:

- If rusty nails are used, will the bulb light up, in comparison to good nails being used (based on observation)?

Explore:

The students will be asked to set up the apparatus as shown below:

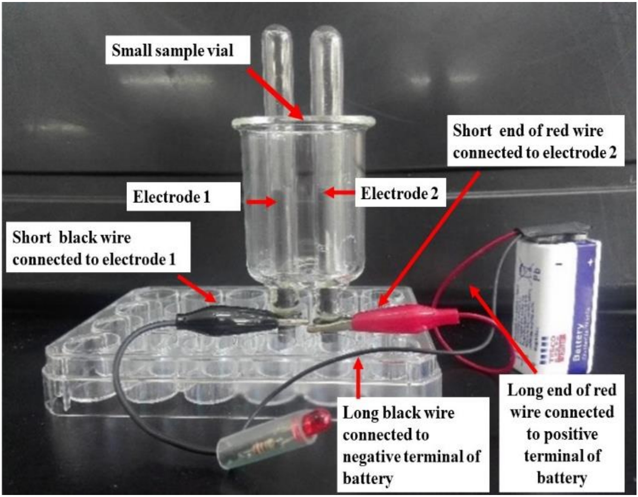
Apparatus:

Combo plate, 9 V battery, current indicator (LED) with wire connections, 2 straw electrodes, 1 straw electrode (with carbon electrode), 1 × carbon electrode (pencil lead), sample vial, box of matches, thin stemmed propette.

Chemicals:

1.0 mol dm^{-3} NaOH and 1.0 mol dm^{-3} $CuSO_4$ solution.

Microscale chemistry set-up:



Procedure:

I. Electrolysis of sodium hydroxide solution

1. Push the current indicator into well E6 of the comboplate.
2. Mark each of the drinking straw electrodes. Let one of the electrodes be as electrode 1 and the other electrode 2.

3. Fill half of the sample vial with 1.0 mol dm^{-3} sodium hydroxide solution. Place the vial into well E5 next to the current indicator in well E6.
4. Hold electrode 1 with the open end upwards and fill the electrode completely with 1.0 mol dm^{-3} sodium hydroxide solution from the propette.
5. Quickly turn electrode 1 the other way up and place it into the solution in the small sample vial. Repeat this procedure for electrode 2. Return any remaining solution in the propette to the small sample vial. Use tap water to thoroughly rinse your fingers free of the sodium hydroxide solution.
6. Connect the end of the black wire from the current indicator to the negative (-) terminal of the battery. Connect the end of the short black wire to electrode 1.
7. Connect the end of the red wire to the positive terminal (+) of the battery. Connect the other end of the red wire to electrode 2.
8. Let the substance produced in electrode 1 be called substance A. Let the substance produced in electrode 2 be called substance B.
9. Record any observation at the anode, cathode and in the electrolytes.
10. Test the gas gathered at the cathode by using a lighted splinter.
11. Test the gas gathered at the anode by using a glowing splinter.

12. Record all the results.

II. Electrolysis of copper (II) sulphate solution

1. Push the current indicator into well E6 of the comboplate.
2. Fill half of the sample vial with 1.0 mol dm^{-3} copper (II) sulphate solution. Place the vial into well E5 next to the current indicator in well E6.
3. Hold the straw electrode (with carbon electrode) with the open end upwards and fill the electrode completely with 1.0 mol dm^{-3} copper (II) sulphate solution from the propette.
4. Quickly turn the electrode the other way up and place it into the solution in the small sample vial. Return any remaining solution in the propette to the small sample vial. Use tap water to thoroughly rinse your finger free of the copper (II) sulphate solution.
5. Place the carbon electrode (pencil lead) into the solution in the sample vial.
6. Connect the end black wire from the current indicator to the negative (-) terminal of the battery. Connect the end of the short black wire to the carbon electrode.
7. Connect the end of the red wire to the positive terminal (+) of the battery. Connect the other end of the red wire to the straw electrode.
8. Record any observation at the anode, cathode and in the electrolytes.

9. Light the match. Carefully remove the straw electrode from the solution, sealing the open end with your finger when it is out of the solution. Bring the electrode very close to the glowing splinter.

Data and observations:

Electrolyte	Observation		
	Cathode	Anode	Change in solution
Sodium hydroxide			
Copper (II) sulfate			

State your findings from the experiment:

Question:

1. For the electrolysis of copper (II) sulphate solution,
   a) Identify the cations and anions.
   b) What are the ions that move to the anode and to the cathode?
   c) Which ions have been discharged at the anode and at the cathode?
   d) Write half equations representing the reactions that occur at the anode and the cathode.

2. Draw a diagram which shows what happens during the electrolysis of dilute sulphuric acid, H_2SO_4. The diagram should show
   a) The ions present in dilute sulphuric acid, H_2SO_4.
   b) The movement of ions to the anode and the cathode.
   c) The discharge of ions at the anode and the cathode and their respective half equations

Explain:

The teacher will explain that:

- Electrolysis is one of the fundamental studies in electrochemistry to study the non-spontaneous reduction and oxidation (redox) reactions at electrodes by applying an electrical energy (from the battery).
- To study the electrolysis process, an electrolytic cell with a two-electrode system is needed.
- An ionic electrolyte must be present in the electrolytic cell during the electrolysis process.
- One electrode must be connected to the positive terminal (anode) and the other electrode must be connected to the negative terminal (cathode).
- Essentially, positive ions (cations) in the electrolyte will move to the cathode. In contrast, negative ions (anions) will move to the anode.
- During the electrolysis process, the cations and anions will accumulate at the cathode and anode respectively over time.
- However, only one cation and anion at the anode and cathode terminal will undergo the redox process at a time.
- Principally, the reduction reaction occurs at the cathode whilst the oxidation reaction occurs at the anode.

- To determine the specific cation and anion that will undergo the oxidation and redox reactions at the anode and cathode, an electrochemical series as shown in Figure 1 must be considered.
- The electrochemical series is a list of cations and anions arranged in an arising order based on their tendency to undergo the respective redox reactions.

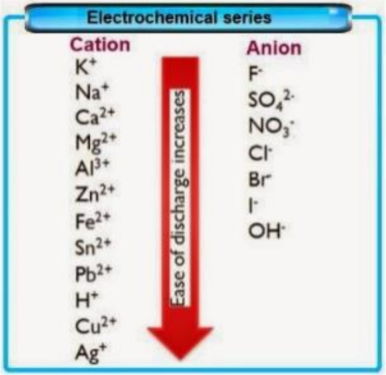


Figure 1: Electrochemical series for cations and anions.

The lower the position of cation and anion in the electrochemical series, the easier the cation and anion to undergo the respective redox reactions. For example, if Cu^{2+} and H^{+} ions are accumulating at the cathode, the Cu^{2+} ions will undergo the reduction reaction. This is because the Cu^{2+} ions are easier to be discharged than the H^{+} ions as the position of Cu^{2+} ions is lower than H^{+} ions in the electrochemical series. The same concept also applies to anions. This will be explained in detail as shown in Figure 2.

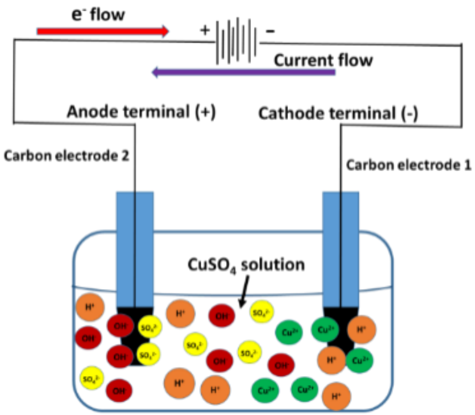


Figure 2: Electrolysis process of blue $CuSO_4$ solution

In the case of the electrolysis of $CuSO_4$ solution, Cu^{2+} ions will move to the cathode whilst SO_4^{2-} anions will move to the anode. In the case of water (H_2O), the H^+ ions will move to the cathode whilst the OH^- ions will move to the anode. Following this process, both anions (OH^- and SO_4^{2-}) are accumulating at the anode. By contrast, both of the anions are accumulating at the cathode. At the cathode, because the position of Cu^{2+} ion in the electrochemical series is lower than H^+, thus, Cu^{2+} ions will undergo the reduction process to form Cu metal at the electrode surface. Meanwhile, at the anode terminal, OH^- ions will undergo the oxidation process to produce oxygen gas (O_2).

The half reactions of the redox processes at the cathode and anode are shown below;

At the cathode:

$$\mathbf{Cu^{2+}(aq) + 2e^- \rightarrow Cu(s)}$$

At the anode:

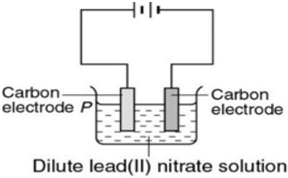
$$\mathbf{OH^- (aq) \rightarrow O_2(g) + 2H_2O (aq) + 4e^-}$$

In both cases, the intensity of the blue colour of the electrolyte decreases as the concentration of blue Cu^{2+} ions in the solution decreases when more copper atoms are deposited at the cathode. Meanwhile, the electrolyte solution becomes more acidic because the H^+ and SO_4^{2-} ions are left in the solution. To test the presence of O_2 in the straw electrode, a glowing wooden splint will be used. The glowing wooden splint will relight if O_2 is collected in the straw electrode.

Elaboration:

The students are asked to do the exercise shown below:

1. The diagram below shows the apparatus arrangement for the electrolysis of the dilute lead (II) nitrate solution.



What is formed at the carbon electrode P?

A. Lead

C. Oxygen

B. Nitrogen

D. Hydrogen

2. When a copper plate is placed into dilute hydrochloric acid, no reaction occurs. This is because

A. Hydrogen is not a metal
B. The mixture needs to be heated
C. Copper is more electropositive than hydrogen
D. Hydrogen is more electropositive than copper

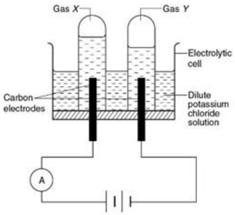
3. Which of the following equations represents the reaction that occurs at the negative terminal of a mercury cell?

   A. $Pb \rightarrow Pb^{2+} + 2e^-$
   B. $Pb^{2+} + 2e^- \rightarrow Pb$
   C. $Zn \rightarrow Zn^{2+} + 2e^-$
   D. $Zn^{2+} + 2e^- \rightarrow Zn$

4. An electrolysis is carried out on solution X using carbon electrodes. If hydrogen is released at the cathode, which of the following is solution X?

A. Copper (II) chloride solution
B. Dilute sulphuric acid
C. Silver nitrate solution
D. Copper (II) sulphate solution

5. The diagram below shows the apparatus set-up that is used to study the electrolysis of dilute potassium chloride solution.



(a) (i) What is meant by cation?

(ii) State the energy change that occurs in the electrolytic process.

(b) (i) Label the ‘anode’ and ‘cathode’ to the diagram above.

(ii) Write the formula of the ions that move to the anode and cathode of the electrolytic cell in the table below.

Anode	**Cathode**

(c) State the observation at the anode and cathode of the cell.

(d) (i) Name gas X.

(ii) Give a chemical test to determine and identify gas X.

(iii) Write a half equation for the formation of gas X.

(e) If 36 cm^3 of gas X is collected at the end of the experiment, calculate the number of molecules of gas X that is collected. [Molar volume = 24 dm^3 at room conditions; $N_A = 6.02 \times 10^{23} mol^{-1}$]

(f) State the ratio of the volumes of gas X and gas Y that are collected.

Evaluate:

The teacher asks a few students to reflect on what they have learned today.

Exp 2 Electrochemistry: Electroplating

Objective:

To investigate the electroplating of an object with copper

Engage:

The teacher shows students the picture of a pewter.

The teacher opens a video that shows a simple process of electroplating.

(https://www.youtube.com/watch?v=gG1HnE8gL9o)

Explore:

The students will be asked to set up the apparatus as shown below:

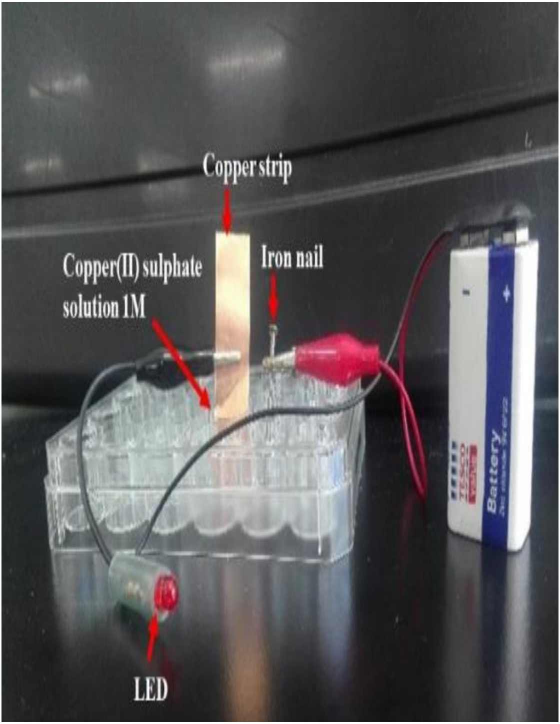
Apparatus:

9 V heavy duty battery, comboplate, current indicator (LED) with wire connections, sample vial and thin stemmed propette.

Chemicals:

1.0 mol dm^{-3} copper (II) sulphate solution, copper electrode/plate, iron nail, sandpaper.

Microscale chemistry set-up:



Procedure:

1. Clean a piece of the iron nail with sand paper. Wash the iron nail with detergent and rinse thoroughly with water.
2. Push the current indicator into well E6 of the combo plate.
3. Remove the lid from a small sample vial and fill half of the vial with 1.0 mol dm^{-3} copper (II) sulphate solution. Place the vial into well E5 next to the current indicator in well E6.
4. Set up the Apparatus: using the iron nail as the cathode and a copper electrode as the anode.
5. Switch the current off after 2-3 minutes.
6. Remove the iron nail from the electrolyte and dry it. Record the change to the iron nail.
7. Repeat steps 1-6 by using copper as the cathode and iron nail as the anode.

Data and Observations

Set	Electrode		Observation
	Anode	**Cathode**	
I	**Copper**	**Iron nail**	
II	**Iron nail**	**Copper**	

Conclusions:

Questions:

1. What three conditions are necessary to electroplate an iron spoon with copper?
2. A good electroplating process is one that results in an even thin layer of coating. Suggest 2 ways on how this can be achieved.

Explain:

The teacher will explain to the students that:

- Electroplating is a process of depositing a layer of metal on another metal by employing the electrolysis technique.
- The process involves passing an electric current through a solution containing an electrolyte.
- This is done by dipping two terminals called electrodes into the electrolyte and connecting them into a circuit with a battery or other power supply.
- The electrodes and electrolyte are made from carefully chosen elements or compounds.
- The biggest advantage of employing the electroplating technique is to protect the electroplated objects from corrosion and to give them an attractive appearance.
- To electroplate a metal onto a different metal
  a) The metal to be plated must be cathode terminal.
  b) The metal at anode must be the pure plating metal.
  c) The electrolyte must contain ions of the plating metal.
  d) The metal to be plated must be clean and free of grease.
  e) The concentration of the ions of the plating metal must be low.
  f) The electric current must be small.

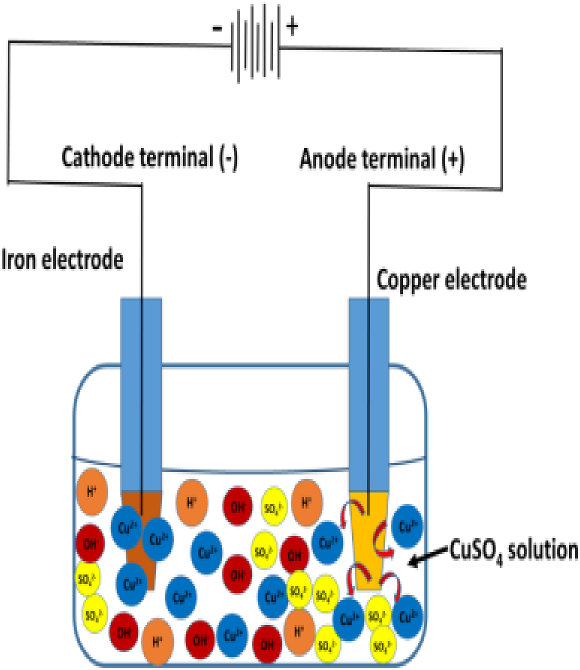


Figure 3: Electroplating process of Cu metal onto the Fe electrode surface.

Based on the figure above, because a pure Cu metal electrode is utilised as an anode, the oxidation process will occur in which the Cu atoms from the Cu electrode will release two electrons to form free-moving Cu^{2+} ions in the solution. Thus, the concentration of Cu^{2+} will increase. From the physical observation, the mass of the Cu electrode will decrease because Cu atoms are already oxidised to Cu^{2+} ions into the solution. To balance the increasing concentration of Cu^{2+} ions in the solution, the Cu^{2+} ions will move to the Fe electrode and then accumulate at the cathode. Because the Fe electrode is used as a cathode, the Cu^{2+} ions will undergo a reduction process to form a layer of Cu metal at the Fe electrode surface. Based on the physical observation, the mass of Fe electrode will increase.

The half reactions of oxidation and reduction at anode and cathode are as shown below;

At the anode:

$Cu\ (s) \rightarrow Cu^{2+}\ (aq) + 2e^-$

At the cathode:

$Cu^{2+}(aq) + 2e^- \rightarrow Cu\ (s)$

Meanwhile, SO_4^{2-} ions (which are negatively charged) will move and will arrive at the positively charged copper anode, releasing electrons that move through the battery toward the negative terminal, Fe electrode. It takes time for the electroplated atoms to accumulate on the surface of the negatively charged electrode. The period of the electrodeposition process depends on the strength of the electric current we apply and the concentration of the electrolyte.

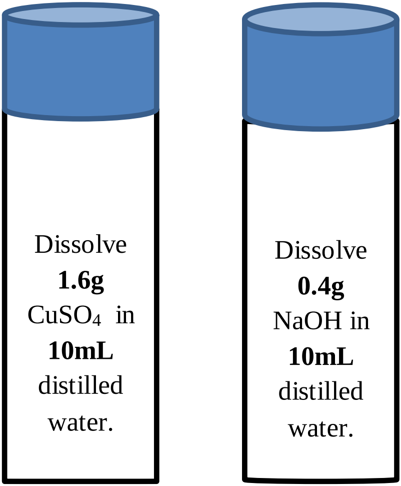
Increasing either one of these parameters increases the speed at which ions and electrons move through the circuit and the speed of the plating process. As long as ions and electrons keep moving, current keeps flowing and the plating process continues.

Elaborate:

The teacher gives the students the situation as shown below and asks the students to solve the problem. Using Diagram of the apparatus set-up and procedure of the experiment, find the observation ion and chemical equation answer and the material and apparatus needs to purify the impure copper plate.

Evaluate:

The teacher will ask the students to give some reflection on what they have learned.



SAFETY PRECAUTIONS

1 mol dm^{-3} $CuSO_4$
Precautions: Use personal protective equipment. Ensure adequate ventilation. Avoid dust formation. Avoid contact with skin, eyes and clothing. Storage: Keep containers tightly closed in a dry, cool and well-ventilated place. Store under an inert atmosphere. Personal Protection: protective eyeglasses or chemical safety goggles, protective gloves, and clothing. Be sure to use an approved /certified respirator or equivalent.

1 mol dm^{-3} NaOH
Inhalation: Not expected to be an inhalation hazard unless it becomes an airborne dust or mist. Skin Contact: CORROSIVE. Eye Contact: CORROSIVE. Ingestion: Can burn the lips, tongue, throat and stomach.

Chemical Safety Data Sheet (SDS)

No.	Chemicals	Link for SDS
1.	1 mol dm^{-3} $NaOH$	https://bit.ly/2G8F8Eh
2.	1 mol dm^{-3} $CuSO_4$	https://bit.ly/2Pou9HO

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