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Copper Recovery of E-Waste Disposal (SIM CARD) by Using Electro-Generated Chlorine

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Abstract— One method for dissolving Cu from e-waste is to use electro-leaching. The waste used is Simcard and NaCl as electrolytes. Simcard contains copper metal and several other valuable metals such as silver (Ag), gold (Au) and palladium (Pa). The electro-leaching process is carried out in an electrolytic cell reactor where chlorine gas will be produced from the electrolysis process, then chlorine gas will extract the copper contained in the Simcard. This research was conducted by varying the electrode potential (voltage)/electric current (amperes), salt concentration and time. From this research, Simcard contains the initial copper (Cu) content of the Simcard was 40.53% and can be separated by electro-leaching method in a stirred acrylic reactor with the best results under operating conditions; a temperature of 30⁰ C, a pressure of 1 atm, a concentration of 10% salt solution, a potential difference of 10 Volts, a leaching time of 60 minutes, and a stirring speed of 600 rpm resulted in a Simcard metal alloy yield of 98.59%.

Keywords—Electro-leaching, Electrode potential, Electric current, E-waste, Simcard

I. INTRODUCTION

The disposal of obsolete and/or defective electronic equipment, called WEEE (waste electric and electronic equipment), in the environment is increasing at the same time. According to the United Nations Environment Program (UNEP, 2006), it is reported that the production of WEEE is about 20 to 50 million tons/year, an amount that is three times that of household waste. One of the components of this electronic equipment is the Subscriber Identity Module Card (Simcard). The basic materials of Simcard are non-metallic materials (such as plastic, fibers, and ceramics) and metals, such as copper, nickel, gold and tin. In general, e-waste consists of ferrous and non-ferrous metals, plastics, ceramics, and other items. Non-ferrous metals consist of metals such as copper, aluminum, precious metals such as silver, gold, platinum, palladium and others. (Neha Lalchandani, 2010). Another factor that makes copper recovery from Simcard attractive is the level of purity; In most cases, the copper recovered from the recovery process is purer than the copper extracted from the ore (Kim et al, 2011).

There are many methods for recovering metals from electronic waste, such as physical and mechanical separation (Chao et al, 2011), pyrometallurgy and hydrometallurgical methods (Kim et al, 2011; Xiu and Zhang, 2010), bio-technology (Zhu et al, 2011).) and others. Of the several processes carried out, the hydrometallurgical process, especially electroleaching, is more efficient for recovering metals from electronic waste (Guimaraes et al, 2014), but the use of leaching agents (such as HCl and H_2SO_4) is still relatively high (Kim et al, 2011), as a result produces wastewater with high pollutant content and needs to be treated for reuse or discharge into the environment. However, this process (by using Electro-generated chlorine) has advantages in terms of relatively low cost and low environmental impact compared to pyrometallurgical processes (Jha et al, 2012). There are 2 reactions that occur in the electrolytic cell, namely the oxidation reaction that occurs at the anode and the reduction reaction at the cathode. In the electrolysis of salt water, a half-cell reaction occurs as below: (http://web.missouri.edu)

Cathode (-) : $2H_2O(l) + 2e^- \rightarrow H_2(g) + 2OH^-$ E^0_{sel}=-0.83 V
Anode (+) : $2Cl^-(aq) \rightarrow Cl_2(g) + 2e^-$ E^0_{sel}=-1.36 V

$$2H_2O_{(l)} + 2Cl^-_{(aq)} \rightarrow H_{2(g)} + 2(OH)^- + Cl_{2(aq)} \quad E^0_{sel} = -2.19\ V \quad (1)$$

As for the complete reaction it would be

$$2H_2(l) + 2NaCl \rightarrow H_2(g) + 2NaOH + Cl_2(aq) \quad E^0_{sel} = -2.19\ V \quad (2)$$

This reaction can occur if E^0_{sel} is positive. In the above equation, E^0_{sel} is negative so it is necessary to conduct electricity of E^0_{sel} = 2.19 V

The next reaction for leaching copper using electro-generated chlorine is as follows:

$$Cu(s) + Cl_2(aq) \rightarrow Cu^{2+} + 2Cl^- \quad (3)$$

$$Cu_{(s)} + Cl_3^- \rightarrow Cu^{2+} + 3Cl^- \quad (4)$$

$$Cu_{(s)} + Cu^{2+} \rightarrow 2Cu^+ \quad (5)$$

Copper dissolves into Cu(I) and Cu(II) chloride complexes; $CuCl_2$, $CuCl^{2-}$, $CuCl_3^{2-}$, $CuCl^+$, $CuCl_2$, $CuCl_3^-$, $CuCl_4^{2-}$ and so on (Lin et al, 1991), where the reaction depends on the potential magnitude and concentration of chloride ions. (Eun-young Kim et al, 2010)

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II. METHOD

II.1. Variable of Research:

1. The voltage used is 5,7.5, 10, 12.5 and 15 volts.
2. Concentration of saline solution 5, 10, 15, 20 and 25%
3. The time used is 30, 60, 90, 120 and 150 minutes

The fixed variables in this experiment are as follows:

- Pressure	1 atm
- Temperature	30°C
- Stirring speed	600 rpm
- Sample	10 pcs SIM card

II.2 Data Analysis and Processing Techniques

II.2.1 Analysis Techniques

The quantities measured during the study were yield and % recovery and filtrate concentration. The concentration of copper (Cu) was obtained from the analysis of Atomic Absorbtion Spectrophotometry (AAS) with standard SNI 06-6989, 6-2004.

III.2.2 Data processing

The processing of data from the results of experiments and AAS analysis uses the following equation:

1. Calculation of metal mass in raw materials

Total mass of metal in SIM card = Initial Mass of SIM card – Final mass of SIM card

2. Calculation of metal yield (%) in the electro-generated chlorine process

$$\text{Yield (\%)}=\frac{(\text{Mass of metal recovered (gr)}}{(Total\ mass\ of\ metal\ in\ SIM\ card)}\ x\ 100\ \%$$

3. Calculation of metal mass reduction on Simcard after electro-leaching process for t minutes:

The mass of the metal on the SIM card = (Mass of the SIM card at minute t1) – (Mass of the SIM card at minute t2)

4. Calculation of the mass of dissolved copper:

Mass of copper in solution = Cu concentration in solution (AAS analysis result) x Volume of solution

5. Calculation of % dissolved copper:

$$\text{\% Copper (Cu) dissolved}=\frac{(\text{Mass of copper in solution})}{(\text{Mass of metal in SIM card})}\times 100\%$$

6. Current density calculation:

$$\text{Current density}=\frac{(average\ current\ strength\ (mA)}{(electrode\ surface\ area\ (cm^2)}$$

II.3 Equipment Used
1. Graphite plate with a surface area of 79 cm2
2. Adjustable power supply
3. Magnetic stirrer and Stirring plate
4. Acrylic reactor as Electrolytic Cell
5. Commercial Polyvinyl Acetal Membrane (Kanebo)
6. Beaker glass 500 mL

II.4 Materials Used
1. SIM card waste
2. Salt (NaCl)
3. Aquades

II.5. Research Procedure

Simcards as many as 10 pcs with size of 12 cm x 12 cm that have been separated are weighed with an analytical balance. The reactor used in this experiment measures 18 cm × 9 cm × 10 cm and is equipped with a magnetic stirrer in the anode compartment. Electrolyte preparation is done by dissolving salt into distilled water according to the desired variable. The electro-generated chlorine process was carried out in an acrylic stirred reactor with a stirring speed of 600 rpm at 30ºC, 1 atm conditions in the reactor. These operating conditions refer to previously reported references (Kim et al, 2011). In the reactor, two processes occur at once, namely the electrolysis process, namely the process of forming chlorine gas and the leaching process where the chlorine gas produced will extract the copper contained in the Simcard.

First, the NaCl solution with a predetermined concentration was put into the electrolytic cell of each compartment as much as 150 mL. Then the adjustable power supply was turned on. Then the voltage was adjusted according to the desired variable. Then 10 samples of Simcard were inserted into the electrolytic cell. The start time of electrolysis was switched on when the adjustable power supply starts running along with stirring with a magnetic stirrer. Chlorine gas formed in the electrolytic cell would react with copper metal to form a $CuCl_2$ solution. Excess chlorine gas was flowed into a 500 mL beaker glass for temporary storage before being discharged. The electro-leaching process was carried out according to the variable potential difference, concentration of salt solution and a predetermined time. The filtrate was taken from the leaching reactor using an injection. The filtrate sampling process was carried out according to the predetermined time variable. The dissolved copper in the filtrate was analyzed using Atomic Absorption Spectrophotometry (AAS).

III. RESULTS AND DISCUSSION

In this research, the raw material used is SIM card electronic waste. SIM card consists of various metals with a metal yield of 42.43%. These results were obtained by dissolving SIM card in a solution of 0.5 M HCl and 2 M H_2O_2. (M. Dimitrijevic, 1998). After dissolved all the metal on the SIM card then weighed with an analytical balance. The solution resulting from the leaching process was analyzed for copper content with AAS (Atomic Absorbtion Spectrophotometry) and obtained a copper percentage of 40.35%.

During the process of electrolysis of salt solution (NaCl), at the anode (positive pole) an oxidation reaction occurred as generation/formation of chlorine gas (Cl_2). This was indicated by a change in the color of the NaCl solution from clear to dark yellow. Chlorine gas can be dissolved in a solution (M. Alkan et al, 2005). The oxidation reaction at the anode is stated as follows:

$$Cl^- \Leftrightarrow Cl_2 + 2e^- \qquad E^0 = 1{,}35\ V \tag{6}$$

The chlorine gas that is formed is then dissolved in water according to the following equation:

$$Cl_{2\,(g)} \Leftrightarrow Cl_{2\,(aq)} \qquad \log K_1 = -1{,}21 \tag{7}$$

$$Cl_{2\,(aq)} + H_2O \Leftrightarrow HCl + HClO \qquad \log K_2 = -3{,}40 \tag{8}$$

$$Cl_{2\,(aq)} + Cl^- \Leftrightarrow HCl + HClO \qquad \log K_3 = -0{,}71 \tag{9}$$

In a salt solution, Cl_2 gas is dissolved as aqueous compounds such as Cl_2 (aq), Cl_3^- and HClO, the distribution of which depends on the concentration of the solution and temperature. (H. Majima et al, 1990; Alkan et al, 2005). The concentration of dissolved chlorine gas increases linearly with time in a 25% saline solution.

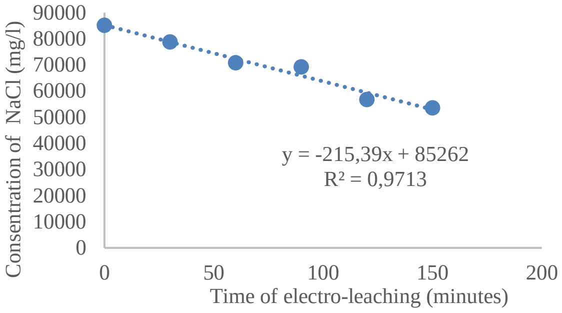

Figure 1. Graph of the relationship between the concentration of NaCl in the reactor (mg/l) with electrolysis time (minutes) at a potential difference of 10 V.

Figure 1 shows a decrease in salt solution concentration during 2.5 hours of electro-generated chlorine process at a salt solution concentration of 25% and a potential difference of 10 V. Initially, the concentration of salt solution before electro-generated chlorine was 85262 mg/l and decreased linearly after 2.5 hours electro-generated chlorine to 53584 mg/l. The decrease in the concentration of this salt solution indicated the process of electrolysis of NaCl into chlorine gas. The distribution of dissolved copper ($CuCl_2$) depends on the potential difference and the concentration of chlorine gas. Chlorine gas produced from the electrolysis process at the anode is soluble in 25% salt solution and is used for the copper leaching process.

At the same time, at the negative pole (cathode) a reaction occurs for the formation of hydrogen gas and the side reaction allows the migration of Cu^{2+} ions from the anode to the cathode through the ion exchange membrane. According to the results of the AAS (Atomic Absorbtion Spectrophotometry) analysis, the weight of copper in the solution increases but when compared to the metal yield, the percent recovery of copper continues to decrease.

III.1 Effect of potential difference on electro-generated chlorine process

Figure 2 shows a graph of the simcard weight loss (mg) over time with a voltage of 5; 7.5; 10; 12.5; and 15 V on electrolysis of 25% salt solution. From the graph it was known that for all voltages, the weight of the SIM card decreased during the electrolysis process. This indicated that there was a reduction in the concentration of metals (mainly copper) in the SIM card. The copper recovery process occured at the anode. (Guimaraes et al, 2014). This decrease indicated that the salt compound (NaCl) could be made unstable and used for leaching various types of metals. This salt (NaCl) was deactivated with the help of electricity to form chlorine gas which was strong enough to dissolve all types of metals.

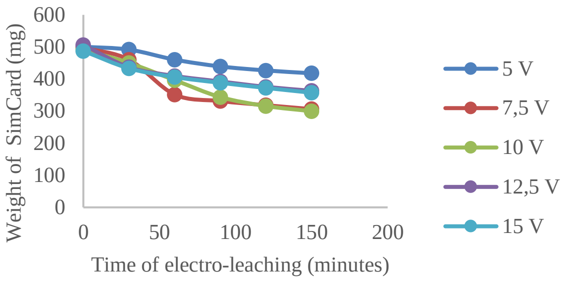

Figure 2. SIM Card weight relationship with time (minutes) on various potential difference variables

At the beginning of the experiment, the metal concentration decreased drastically. This indicated that an oxidation reaction occured on the surface of metal particles (Oishi et al, 2007). This drastic weight loss was due to the nature of copper metal which was easily corroded. (Eun-young Kim, 2011). Experimental variables were limited to 150 minutes because the decrease in mass decreased drastically in the first 60 minutes so the experiment was not continued. In addition, dissolved chlorine shows its saturation point at 150 minutes (Eun-young Kim, 2011). At a high potential difference (> 10 Volts) chlorine gas would formed quickly and corroded copper metal quickly, in proportion to the amount of chlorine gas formed. The higher the voltage, the faster the leaching rate and the reduced residence time in the compartment. But the high voltage had a disadvantage, namely the rate of chlorine gas that was formed too quickly so that it affected the life of the electrode. At high voltages the electrode would be damaged and a layer was formed that inhibited the rate of electric current. This was evidenced by the decrease in current that continued to decrease during the leaching process at a voltage of 12.5 v and 15 v. If the current flowing decreases, the chlorine gas produced would also decrease. Decreased chlorine gas would reduce the leaching rate which had an impact on reducing the % recovery of the Simcard.

Figure 3 shows the relationship between potential difference and % metal yield in the electro-generated chlorine process. Yield percentage of potential difference variation in the 2.5 hour electro-leaching process, namely; 5 Volt was equal to 38.92% ; 7.5 Volts of 90.26% ; 10 Volts of 90.30% ; 12.5 Volts at 67.2% and 15 Volts at 61.22%.

The optimum conditions for the electro-generated chlorine process shows at a voltage of 7.5 volts to 10 volts. At this voltage the current flows constantly and the chlorine gas produced was also constant. The constant rate of chlorine gas made the performance of the carbon electrode stable and the leaching rate could be maintained constant. So that the optimum potential difference for the electro-leaching process with a salt solution was 10 V with a yield percent of 90.30%.

Based on Figure 3, the optimum point was found at 10 V. This was because the Simcard weight loss rate tends to be constant. This constant decrease also indicated that the electrode was not damaged and no passive layer was formed. This was evidenced from Figure 4 where the current flowing tends to be constant so that the rate of chlorine gas formed also tends to be constant. In addition, at this voltage, optimum results were obtained based on the dissolved metal yield.

But at a voltage of 10 V-12.5 V there was a decrease in % yield from 90.30% to 67.2%. This was due to a decrease in the performance of the carbon electrode. According to Rabah (1991), the greater the current density in the electrolytic, the faster the graphite oxidizes, thereby reducing the performance of the carbon electrode. The carbon electrode would formed a thin layer which could reduce the current distribution so that it had an impact on decreasing the chlorine gas produced. If the chlorine gas formed was getting smaller, the metal yield would also decrease proportional to the chlorine gas produced. The greater the voltage, the current density would also increase. The increasing current density affected the chlorine gas produced so that the leaching rate wouldl increase. But a large voltage had a negative impact on the electrodes. The electrode would form a layer that could inhibited the electric current so that a high voltage was not good for the leaching process because the leaching rate reached its maximum at the beginning of the process and continued to decrease over time.

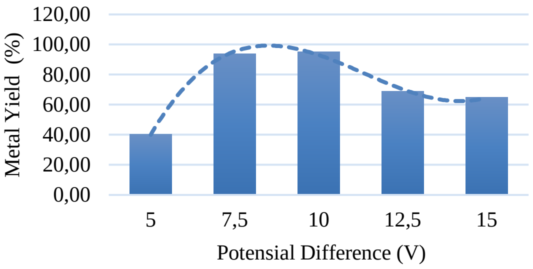

Figure. 3. Graph of voltage relationship with % metal yield on SIM card in 25% salt solution

In this experiment, observations of current strength fluctuations were also evaluated. This was considered important because the strong current density helped the mobility of copper ions (Nunez et al, 2013). Changes in electric current were observed at potential differences of 5, 7.5, 10, 12.5, and 15 V every 30 minutes (Figure 4). Electric current tends to be stable at potential differences of 5, 7.5, and 10 V, while at potential differences of 12.5 and 15 V there was a drastic decrease. This was due to the presence of chloride ions which were oxidized to chlorine gas. The formation of chlorine gas was very fast because more and more chloride ions were oxidized and the electrical conductivity of the electrolyte ions became small, resulting in a decrease in electric current. (Kristanti et al, 2013). Besides that, there was a passive layer that blocked the flow of electric current so that the electric current would decrease.

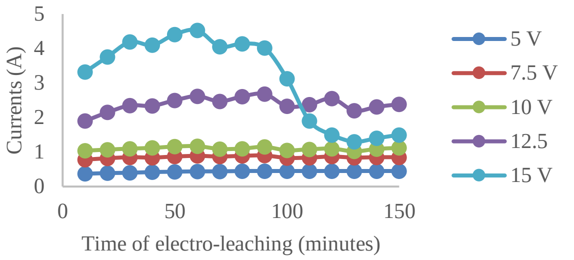

Figure 4. Relationship of time to current at various voltages

III.2 Effect of salt concentration on electro-generated chlorine

Figure 5 shows the percent Yield as a function of the concentration of the salt solution with a range of 5% - 25% at a temperature of 30ºC and a potential difference of 10 V. The most substantial reduction in Simcard weight during the 60 minutes electro-generated chlorine period was in electro-generated chlorine with a solution salt 10%. So that the most optimum concentration of salt solution (NaCl) for the electro-generated chlorine process was in a 10% salt solution with a yield percentage of 98.59%. Factors that influenced the occurrence of this was the pH value. The pH value was very important to consider in the leaching process using Chlorine Gas because the chemical nature of Chlorine gas depended on the pH value (H. Majima et al., 1990). Kim et al. (2011) stated that when the pH value was below 2, the dominant compound from chlorine gas was Cl_2 (aq), Cl_3^- while when the pH was above 2 HClO was the dominant compound. The greater the concentration of the salt solution, the HClO distribution process decreases and would run out.

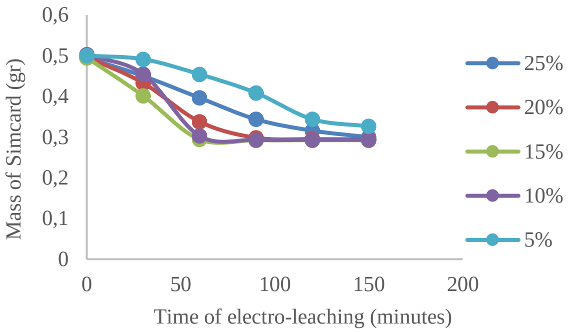

Figure 5. The relationship between electro-generated chlorine and simcard mass at various concentrations of salt solution

In this experiment the optimum time for the electro-generated chlorine process was also evaluated. This optimum time was obtained when metals other than Cu such as Zn, Ni, Al, Pb, Sn and Fe had also been recovered. This was evidenced by the absence of a decrease in the mass of the Simcard if the electro-leaching process was continued.

Figure 6 shows a graph of the decrease in SIM card mass against electro-leaching time using 10% salt solution. In the first 30 minutes, the decrease in metal mass was not too high, from 0.210 g to 0.161 g. Meanwhile, a sharp decrease in SIM card mass occurred at the time of electrolysis between 30 and 60 minutes, from 0.161 gr to 0.010 gr. These results were in accordance with previously reported studies. (Guimaraes et al, 2014). After the electro-generated chlorine process for 60 minutes, all metals have been recovered. So that the optimum time for processing electro-generated chlorine using a saline solution was 60 minutes.

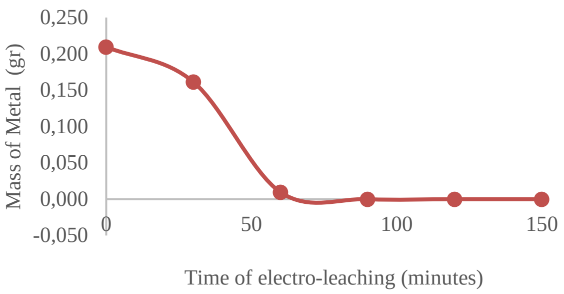

Figure 6. Metal mass reduction with time electro-generated chlorine using 10% salt solution at 10 V potential difference

Figure 7 shows a graph of the percent yield against variations in the concentration of salt solution. At a concentration of 5% salt solution, the percent yield (metal recovery) that occurs was 81.84% and then increases drastically at a concentration of 10% with a percent yield of 98.59%. However, at concentrations >10%, the percent yield actually decreased to 95.44% for a 15% salt solution concentration, 95.15% yield percent for a 20% salt solution concentration and 91.81% yield percent for a 25% salt solution. So that the optimum concentration of salt solution for the electro-generated chlorine process was obtained, namely at a concentration of 10% salt solution.

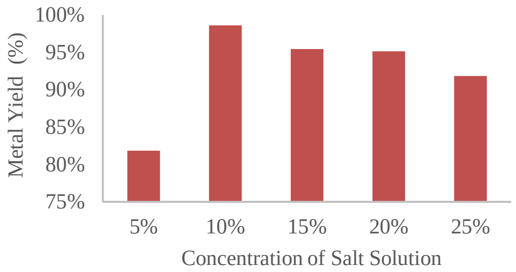

Figure 7. Relationship between concentration of salt solution and percent Metal yield

Figure 8 shows a graph of the solubility of chlorine gas in the electro-generated chlorine process for 2.5 hours in a 25% salt solution and a potential difference of 10 V. In a salt solution, Cl_2 gas was dissolved as Cl_2 (aq), Cl_3^-, and HClO compounds. Alkan et al., (2005) stated that the distribution of these compounds depends on the concentration of the solution and temperature. From the graph it was known that the concentration of dissolved chlorine gas increases linearly with time in a 25% salt solution. This was in accordance with previous studies where the concentration of dissolved chlorine gas increased in other electrolysis solutions such as HCl solution (Kim et al, 2011).

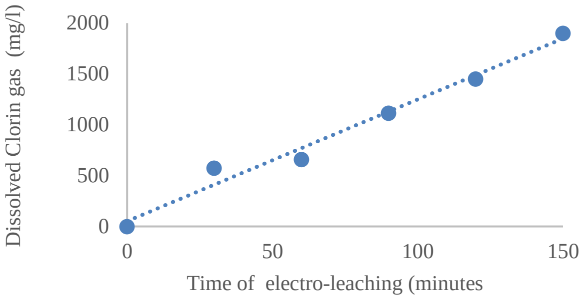

Figure 8. Relationship of dissolved chlorine gas to time electro-generated chlorine with a salt solution concentration of 25% and a potential difference of 10 V

IV. CONCLUSION

1. Recovery of copper metal (Cu) from Simcard electronicwaste (Cu content 40.53%) can be done by electro-generated chlorine process using a salt solution (NaCl) with a Yield percentage of 98.59%.
2. The optimum potential difference for the electro-generated chlorine process using a salt solution is 10 V
3. The optimum concentration of salt solution (NaCl) for the electro-generated chlorine process is 10% salt solution.
4. The optimum time for the electro-generated chlorine process using a salt solution is 60 minutes

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