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Concentration Profile Behavioral from Digestate Television Printed Circuit Board for Metal Recovery via Electrolysis

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Abstract: The production of electric and electronic products is one of the fastest growing businesses in the world. This scenario creates increasing amount of electric and electronic waste in our waste stream and it becomes a common problem facing by the world. In this study, the concentration profile behavioral of a television printed circuit board (PCB) is being investigated for future electrolysis treatment. Prior to that, a PCB is cut into several pieces and is digested via aqua-regia. The digestate from the digestion is analyzed using Inductively Coupled Plasma (ICP-MS) to estimate the present of metals with their range of corresponding concentration value and Thermogravimetric Analysis (TGA) is carried out to investigate the thermal decomposition of the filtrate from the digestion. The major metal elements that contain in the digestate are Cu (3,123-6,994 mg L^{-1}), Pb and Fe and traces of two precious metal namely (6.3-13.8 mg L^{-1}) and Au (0.2-5.4 mg L^{-1}). Three degradation stages are observed in the TGA analysis (two days after ovenate at 102°C) namely moisture removal with 10.3 wt %, volatile matters removal with 14.45 wt.% and non-volatile removal with 16.7 wt%. The ash value after 1000°C heating in the TGA is almost 60 wt. %. An electrolysis process is then proposed in order to recover selectively valuable metals, such as Cu, Pb, Ni, Ag and Au.

Key words: E-waste, aqua-regia, electrolysis, metal recovery

INTRODUCTION

Electronic products that are near or at the end of their useful life are referred as e-waste. Traditionally, electronic products have been considered durable items where owners have retained them over many years, even decades. As a real cost for electronic products decreased, and as the economy has grown, electronic products have been replaced with increasing frequency. The consequence is a burgeoning quantity of electronic product waste that poses a significant and expensive management challenge to be faced on which correlated with human health and environment (APHA, 2006). A total of 53 thousand metric tonnes of electronic waste from obsolete computers and other electronics are being generated in Malaysia in 2007 and electronics waste already constitute 4.6% of the Malaysian scheduled wastes and it keeps growing rapidly (DOE, 2007). The aim of this study is to investigate the concentration profile behavioral of a Television Printed Circuit Board (PCB). PCB are typically made from reinforced polymeric materials and range in cost and quality from the low cost paper filled phenolic materials found in consumer electronics through the glass fibre reinforced epoxide based laminates, to more expensive higher performance materials which use high temperature resistant, chemically and thermally stable polymers such as polymides and PTFE. A typical PCB will have metallic conductor that are formed by etching of patterns in a laminated copper foil structure. In the simplest PCBs the copper will be the only structure on the board and it is thus known as a single-sided board; more sophisticated boards may be double-sided and multi-layered (Coosey and Keller, 2002). Depending on the board design, the copper could typically cover 25% of the circuit board area and would vary in thickness from less than 20 microns to more than 200 microns if the conductors need to carry a significant current (Legarth *et al*., 1995). In this study the concentration profile behavioral of a television printed circuit board (PCB) is being investigated for future electrolysis treatment.

MATERIALS AND METHODS

Material: Nitric Acid (65%) Analytical Reagent HmbG Chemical, Hydrochloric Acid (37%) Analytical Reagent HmbG Chemical, Hydrogen Peroxide (30%) Analytical Reagent Grade Fisher Scientific from United Kingdom and

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Distill water were used for digestion procedure. Digestion vessel Analytical Grade Fisher Scientific, Glass fiber filter Advantec grade GD-120 (47 mm) from Japan and N-WW EYELA Rotary vacuum water pump evaporator from Japan were used for filtration of digestate. Multiparameter Inolab WTW from German were used for pH value identification of the digestate. HI 8733 Conductivity meter HANNA Instrument from Italy were used for obtain conductivity detection of the digestate.

Experimental procedure: Televisions PCB were obtained from a retail shop. In this study, the simplest PCBs were used which also known as single-layer PCB. The PCB was digested using a combination of two kinds of acid which is nitric and hydrochloric acid, where the combination of these acids is known as Aqua Regia solution. There are many kinds of volume ratio arrangement used in digestion process in the industry but for this research work combination of 3:1 (HNO_3: HCL) volume ratio were used. Prior to each digestion, the board were shredded into smaller pieces where this is often primarily undertaken to reduce the bulk size of the board and also to expose a greater surface area of the board to contact with the aqua-regia. The time duration of single batch digestion process would normally take around five hours to be completed. The digestion process implemented throughout this research follows a standard method ASTM 3050B. Completion of digestion process means that all the metallic fractions contained within the scrap board were completely dissolved in the aqua-regia. The digestate were then filtered using glass fiber filter assisted with water pump to separate the filtrate and the solid residue for further analysis. The filtrate would be analyzed with three analyses, namely conductivity, pH and metal concentration analysis; the solid residue would undergo thermal decomposition analysis.

RESULTS AND DISCUSSION

Digestion: Sample around 20 g of shredded Television PCB were introduced for each batch of digestion process. Each digestion process utilized 300 mL of nitric acid, 100 mL of hydrochloric acid, 100 mL of distill water and 50 mL of aliquots consists of 30 mL of hydrogen peroxide and 20 mL of distill water. There were total of 10 batches of samples were digested for shredded television PCB. Table 1 illustrates the weight of samples for the total of 10 batches being dissolved in the digestion process.

Based on Table 1, it is found that the average value for the weight loss percentage of the sample is calculated as 63.8%. The value given for the after digestion sample weight were taken after the residue being filtered and leaves in the furnace ovenated at temperature of 102°C for two days of time. There was some portion of the solution being vaporized due to the heating during the digestion process. The average percentage of solution volume being vaporized during the reaction process to the total volume of solution being set up for the system was calculated as 33%.

Inductively Coupled Plasma (ICP): Process of digestion using aqua-regia is also known as hydrometallurgical chemical treatment. There are two kinds of approaches to recover metals from electronic PCB scrap either using selective or non-selective dissolution method (Oh *et al*., 2003). In this study, non-selective dissolution method was used, where this method are able to dissolve all kind of metals that present in the PCB scrap, whereby using selective dissolution method, only able to dissolve individual metal. Table 2 tabulates the metals concentration in mg L^{-1} of the filtrate from the digestion process.

The dissolved metals generated via chemical dissolution from the scrap PCB are presented as ionized species within the aqueous media. Analysis of ICP was used to estimate the present of metals with their range of corresponding concentration value. Referring to Table 2, the major metal elements that contain in the solution are Cu (3,123-6,994 mg L^{-1}), Pb (446-533 mg L^{-1}) and Fe (1,177-2,075 mg L^{-1}) and two precious metals are traced namely Ag (6.3-13.8 mg L^{-1}) and Au (0.2-5.4 mg L^{-1}). The values of the analysis for each batch were varied which

Table 1: Total weight dissolve via digestion

	Weight of sample (g)			
Batch No.	Before digestion	After digestion	Weight dissolves (g)	Weight loss (%)
1	20.400	6.847	13.553	66.44
2	20.100	6.616	13.504	67.31
3	20.400	5.212	15.189	74.46
4	20.270	7.502	12.770	63.00
5	20.720	6.379	14.340	69.21
6	20.400	5.932	14.468	70.92
7	20.950	9.555	11.400	54.42
8	20.330	7.770	12.560	61.78
9	20.350	10.550	9.800	48.16
10	20.900	7.960	12.940	61.91

Table 2: Concentration of metals constituent (mg L^{-1})

Batch No.	Ca	Cu	Fe	Ni	Pb	Zn	Ag	Au
1	78.0	5,261	2,001	15.7	462.3	22.3	8.3	1.7
2	269.3	3,589	1,821	23.6	460.3	11.3	6.7	0.4
3	19.9	4,264	1,905	61.4	468.0	24.2	8.5	0.4
4	47.0	3,384	1,644	22.5	468.0	40.9	13.8	-
5	34.9	4,519	1,913	88.0	533.3	196.7	13.2	1.1
6	60.5	4,773	1,177	46.4	527.7	70.8	11.4	-
7	70.8	4,813	2,075	515.7	488.3	487.7	7.4	0.9
8	39.2	6,994	2,034	64.6	607.0	63.6	6.3	5.4
9	289.0	4,070	1,372	34.2	446.7	787.3	7.6	0.9
10	52.4	3,123	1,518	658.7	465.3	13.6	10.2	0.2

Table 3: Conductivity and pH value of the diluted filtrate

Batch No.	Conductivity (μS)	pH
1	27.2	1.39
2	29.2	1.35
3	27.6	1.33
4	33.7	1.29
5	26.0	1.43
6	27.3	1.41
7	25.5	1.31
8	27.1	1.44
9	31.1	1.29
10	25.6	1.44

Table 4: Thermo gravimetric analysis result summary

		Temperature zone (°C)						
		(50-150)		(200-600)		(700-1000)		
Batch No.	Original weight (mg)	Weight loss (mg)	Percent of loss (%)	Weight loss (mg)	Percent of loss (%)	Weight loss (mg)	Percent of loss (%)	Ash remain (%)
1	11.2	0.86	7.65	6.60	58.72	1.10	9.79	23.84
2	12.0	1.02	8.50	5.05	42.08	2.80	23.33	26.08
3	12.3	0.73	5.92	5.03	41.08	1.56	12.77	40.24
4	14.6	0.58	3.97	6.01	41.16	2.72	18.63	36.23
5	10.4	0.47	4.52	4.96	47.69	2.26	21.73	26.06
6	11.0	0.44	3.95	6.79	61.56	2.00	18.13	16.36
7	13.8	1.31	9.50	7.28	52.79	1.30	9.43	28.28
8	11.1	0.54	4.89	6.91	62.53	1.40	12.67	19.91
9	13.4	0.70	5.22	6.40	47.76	2.30	17.16	29.85
10	11.6	0.80	6.90	7.30	62.93	1.70	14.66	15.52

might due to the different samples containing small chips taken after shredded for digestion or process sensitivity of the instrument. The present of copper in large amount are already been expected, as a single-sided board was used for the digestion process.

Conductivity and pH: The values of conductivity of the filtrate are important for the future works of this research, where by gaining the conductivity value; it is easy to manipulate the voltage and amount of electricity required for electrowinning process. The higher conductivity value represents the higher excites ability of particles in the solution to move through the solution which is important to determine which elements will be captured during electrowinning (Jennifer, 2002). Table 3 shows the pH and conductivity values for the filtrate.

Table 3 shows that the average conductivity value of the solution is calculated as 28 μS while the pH value varies between (1.29-1.44). Due to the usage of highly concentrated acid during the digestion process, the value of pH and conductivity of the pure filtrate are not able to be detected by the instrument used, further dilution of the filtrate with distill water were proceeded to gain the respective value.

Thermo Gravimetric Analysis (TGA): Residues from the filtration were analyzed using TGA instrument to determine the possible weight loss of sample on heating as a function of time. It is also commonly utilized to analyze the characteristic of sample degradation temperature and absorbed moisture content of the sample. Around (10-15 mg) of residue sample from each batch were analyzed. Table 4 shows the thermal degradation profile for the residue of each batch.

Table 4 shows that there were three degradation stages occurred during the analysis. The first degradation

curve was found in the temperature range of 50-150°C and an average weight loss about 6% from the total weight of samples. This degradation is due to the moisture losses of the sample. As can be seen from Table 4, there is a second degradation occurs at the temperature range of 200-600°C and an average weight loss about 52% from the total weight of the samples. The final degradation occurs at a temperature range of 700-1000°C and an average weight loss about 15% from the total weight of the samples. There were two region of degradation occurs at the temperature range of 200-1000°C; it is generally because of brominated epoxy resins are thermally less stable than nonbrominated ones (Luda *et al*., 2002). This may cause the thermal degradation temperature shift to a lower temperature for the brominated epoxy resins.

According to the literature concerning the thermal decomposition of brominated epoxy resins, the initial reaction is related to the presence of an amine group (good attacking nucleophiles) and bromine atoms (good leaving group). The first stage reaction forms C-N bonds and blocks aromatic amines from escaping to the gas phase. Therefore, the aromatic amines and tar are volatilized during second-stage pyrolysis (Balabanovich *et al*., 2004; Nakao *et al*., 1989; March, 1992). The thermoplastic and thermosetting plastic also one of the reason for the existence of two stages of decomposition, where the thermoplastic substance will volatile at temperature lower than compare to the thermosetting plastic substance.

There was about an average of 26% of ash remaining in the crucible after the heating process completed, where this value represent the fixed carbon contained in the samples.

CONCLUSION

The study presented here described the concentration profile behavioral of the Television PCBs, where it could be concluded that the major element that contain in the PCB are mainly copper, iron and lead. The presences of small amount of precious metal were also traced in this work namely gold and silver. Three degradation stages were observed during the thermal decomposition analysis. The high conductivity values of the filtrate display the viability of utilizing electrowinning process in the future work to recover metal elements.

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