

Article

Leaching and Adsorption of Gold from Waste Printed Circuit Boards Using Iodine-Iodide Solution and Activated Carbon

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Abstract. Iodine-iodide leaching and activated carbon adsorption processes for recovery of gold (Au) from waste printed circuit boards (WPCBs) were discussed in this paper. A pressure oxidative acid leaching (POAL) was carried out in an autoclave using diluted sulfuric acid (1 M H₂SO₄) solution in order to remove high concentrations of some base metals especially copper (Cu), aluminum (Al), iron (Fe) and zinc (Zn) due to their negative effects on gold dissolution from WPCBs. The factors affecting the performance and efficiency of the iodine-iodide leaching process; such as iodine/iodide concentration, pulp density, leaching time and leaching temperature were optimized in order to maximize the gold dissolution efficiency from the WPCBs in the iodine-iodide solution. Results indicated that the vast majority (> 99 %) of gold was dissolved in the solution from the WPCBs under the optimized leaching conditions. Adsorption tests were conducted on leach liquor solution resulting from the iodine-iodide leaching using activated carbon. Nearly 98 % of gold was adsorbed from the liquor solution onto the carbon under the conditions optimized in this study. The results obtained revealed that gold can be successfully recovered from this secondary resource, where the percent recovery amounts to nearly 97 % for gold.

Keywords: Gold, iodine-iodide, activated carbon, high pressure oxidative leaching.

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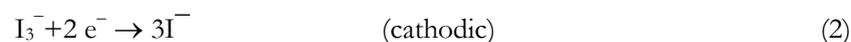
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1. Introduction

Printed circuit boards (PCBs) are essential parts of most electrical and electronic equipment (EEE). The demand for production of EEE is increasing year over year due to rapid economic growth and development of science and technology. Correspondingly, the amounts of waste printed circuit boards (WPCBs) are dramatically increasing as a result of the growing amount of discarded waste electrical and electronic equipment (WEEE), which can not only be a source of composite materials (filler) but also a source of valuable metals such as Cu, Au, Ag, Pt and Pd. The amounts of valuable metals in WPCBs are several times higher than that of naturally occurring minerals [1-3]. Therefore, recycling of WPCBs is an important process to reduce the amount of disposed hazardous wastes and to promote the recovery of valuable metals. There have been several studies on the recycling of WPCBs by using pyrometallurgical, hydrometallurgical, bio-hydrometallurgical and physical-mechanical processes [4-6]. However, an efficient and environmentally friendly technologies are needed to recycle these wastes. In hydrometallurgical process, reagents like hydrochloric acid (HCl), nitric acid (HNO₃), sulfuric acid (H₂SO₄) and hydrogen peroxide as oxygen source were widely used for dissolution of base metals from the waste. Alkaline solution of cyanide is the most used lixiviant for extraction of gold from ores and secondary resources [7, 8]. The Merrill-Crowe zinc precipitation and adsorption onto activated carbon are main processes for gold recovery from the pregnant cyanide solution containing the dissolved gold. In addition, many other separation methods such as co-precipitation, adsorption onto various solid- and bio-sorbents, ion exchange, and solvent extraction have been used for this purpose [9-13]. The utilization of cyanide in the gold leaching process presents risks to environment and health, therefore an economically viable and selective method while minimizing the risks is a very important to develop a process for gold recovery. Thiourea, thiosulfate and halides (chlorine, bromine and iodine) as alternative lixiviants to cyanide can be used for the recovery of gold from natural and secondary sources [7, 8, 14]. Among them, iodine forms the most stable complex in aqueous solution compared with other lixiviants due to the lower standard redox potential ($E^0_{AuI^+} = 0.53$ V) of its complex with gold. A number of researchers have studied this leaching system and they illustrated the mechanism of gold dissolution in an iodine-iodide solution as below [15, 16]:



The solution chemistry of the gold-iodide system is very complicated due to the formation of many different iodine and iodide species in the solution. However, many researchers have been studied the fundamentals of gold recovery from pregnant gold-iodide aqueous solution using activated carbon [17], ion-exchange [18] and precipitation [19], detailed information about this complete gold recovery process is still insufficient. Accordingly, more works and studies are needed to clarify this complicated process.

The aims of this study were the optimization of the processes for gold recovery from WPCBs by using iodine-iodide leaching and activated carbon adsorption. The adsorption of gold on activated carbon is one of the common procedure for the recovery of gold from cyanide solutions, but it has not been used widely for the processing of gold-iodide solutions. The most important variables for gold extraction from WPCBs in iodine-iodide solution and gold adsorption from the pregnant liquor onto activated carbon were studied and determined the optimum extraction and adsorption conditions, respectively.

2. Experimental

2.1. Materials

Waste printed circuit boards (WPCBs) collected from discarded computers were used in this study. Reagents grade chemicals used in this study were iodine, potassium iodide (KI), hydrochloric acid (HCl), nitric acid (HNO₃), sulfuric acid (H₂SO₄), soluble starch, sodium thiosulfate (Na₂S₂O₃), sodium chloride (NaCl) and sodium hydroxide (NaOH). Distilled water was used to prepare all solutions. Granular activated carbon (Nakarai chemicals, Ltd, Japan) was ground and separated on designated sieves for size classification

in order to utilize it in gold adsorption study. The contents and components of various solid residues after completed the processes (incineration, leaching and adsorption) were determined using X-ray Diffraction (XRD), RINT-2200/PC (Rigaku) and X-ray Fluorescence (XRF), ZSX Primus II (Rigaku). Concentrations of ions in the aqueous phases obtained from leaching, extraction and elution were measured by inductively coupled plasma optical emission spectrometer (ICP-OES), SPS-5500 (Seiko Instruments Inc).

2.2. Methods

2.2.1. Pretreatment Processes (Incineration and Pressure Oxidative Acid Leaching)

Waste printed circuit boards (WPCBs) collected was shredded around 2-3 cm in size and incinerated at 800 °C into a furnace (DOWA Metals & Mining Company, Japan) equipped with gas cleaning systems in order to remove organic contents and volatiles. The WPCBs sample/ash obtained from incineration was milled and solid particles passed through a 106 µm sieve was used in the next pretreatment process. A pressure oxidative acid leaching (POAL) was performed using an autoclave under the previously determined conditions which were the concentration of H₂SO₄ of 1 M, pulp density of 100 g/L, partial pressure of oxygen of 2 MPa at 120 °C for 30 min. The autoclave was equipped with 1.0 L reaction vessel, impeller, inlet and outlet gas pipe, pressure and temperature sensors, and electrical heating system.

2.2.2. Iodine-Iodide Leaching

The experiments for gold leaching from the WPCBs ash sample in iodine-iodide solutions were conducted in a 0.5 L baffled volumetric flask immersed in a thermostatically controlled water bath. Agitation was done by a flat blade impeller. Effects of various important variables such as iodine/iodide concentration, pulp density, contact time and temperature on the kinetics of gold dissolution from the WPCBs sample in the iodine-iodide solution were investigated in order to optimize this process.

2.2.3. Gold Adsorption onto Activated Carbon

Activated carbon used in the adsorption was first washed with 2 M HCl followed by hot distilled water and dried at 70 °C for 24 h. Physical characterizations such as surface area, pore size and pore volume of the activated carbon were measured by Quantachrome instruments (Autosorb iQ/ASiQwin) using nitrogen adsorption-desorption isotherm. The carbon samples were degassed at the temperature of 300 °C for 3 h before measurement. An appropriate amount of the activated carbon was added to the pregnant solution resulting from the iodine-iodide leaching into a stoppered conical flask and the mixture was agitated using a thermostatically controlled shaking bath (EYEL 4, NTS-400, Rikakikai). Different adsorption parameters like gold concentration, iodine/iodide concentration, particle size of activated carbon, carbon dosage, pH of solution and shaking speed, conduct time and temperature were investigated. The efficiency (η) of gold adsorption onto activated carbon was calculated by the following equation (Eq. 4).

$$\eta = \frac{C_o - C_{ads}}{C_o} * 100\% \quad (4)$$

where C_o is the initial gold concentration into the pregnant gold-iodide solution (ppm), C_{ads} is the concentration of gold in the raffinate (ppm).

2.2.4. Consumption of Iodine in Leaching and Adsorption

The iodine consumption in the processes of gold leaching and gold adsorption was evaluated by a redox titration method which is based on the oxidation of iodide into iodine in the presence of sodium thiosulfate and starch indicator [20, 21]. The redox reaction in the solution can be illustrated as below:



3. Results and Discussion

3.1. Pretreatment processes

3.1.1. Incineration (800 °C) and Pressure Oxidative Acid Leaching (POAL)

The chemical contents of the WPCBs samples before/after incineration and after POAL are summarized in Table 1 and XRD patterns of the samples are shown in Figure 1. Results showed the contents of the most elements exist in WPCBs sample were upgraded after the incineration process due to the release of volatile materials. As a result of POAL process, the contents of precious metals especially gold (Au) and silver (Ag) are at least 2 times higher, while the amounts of the other accompanying elements (Cu, Fe, Al and Zn) are several times lower compared to the sample after incineration at 800 °C. Table 1 shows the accompanying elements especially Cu and Al are dissolved efficiently from the incinerated WPCBs sample into a 1M H₂SO₄ solution by the POAL process.

Table 1. Chemical contents of the WPCBs samples.

Pretreatment	Elements													
	Au	Ag	Pt	Pd	Cr	Co	Mn	Cu	Fe	Al	Pb	Zn	Ni	Ca
	g/t							wt%						
before incineration	230	480	63	170	1700	230	740	13.4	1.3	2.8	1.8	0.9	0.4	4.3
after incineration	278	1494	107	112	977	230	650	34.4	1.9	3.8	1.6	5.1	2.9	4.6
after POAL	458	2504	68	209	799	240	171	0.24	0.7	0.7	2.5	0.2	0.5	2.1

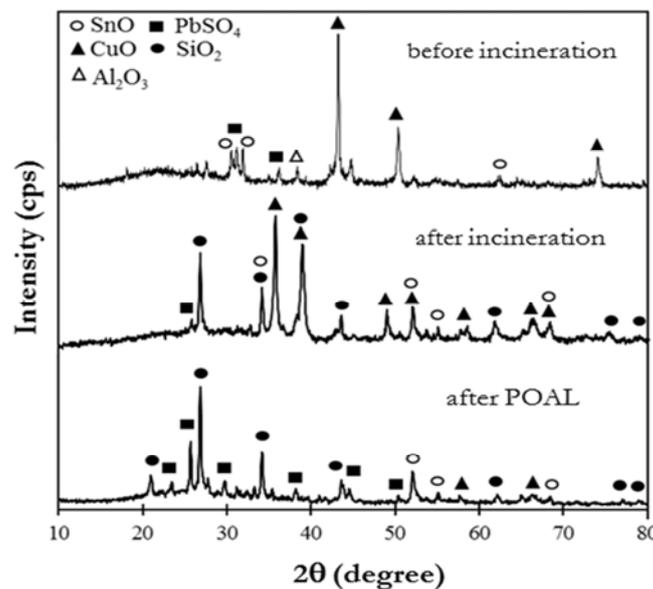


Fig. 1. XRD patterns of the WPCBs samples before/after incineration and after POAL.

XRD patterns for the samples revealed that main constituents of the WPCBs samples are oxides of base metals (Cu, Al, Sn and Pb) and quartz (SiO₂), and the XRD peak intensities of copper oxide (CuO) and aluminum oxide (Al₂O₃) are decreased and disappeared after the sequential pretreatment processes. It was confirmed that the vast majority of Cu and Al are dissolved in a diluted H₂SO₄ solution.

3.1.2. Preliminary Iodine-Iodide Leaching

Preliminary iodine-iodide leaching experiments were conducted using the samples before and after POAL process under same leaching conditions and the leaching efficiency of gold from the samples in iodine-

iodide solution is shown in Figure 2. The results indicated that the maximum efficiency of gold dissolution from the samples before and after POAL in the iodine-iodide solution is 15 % and 99 %, respectively.

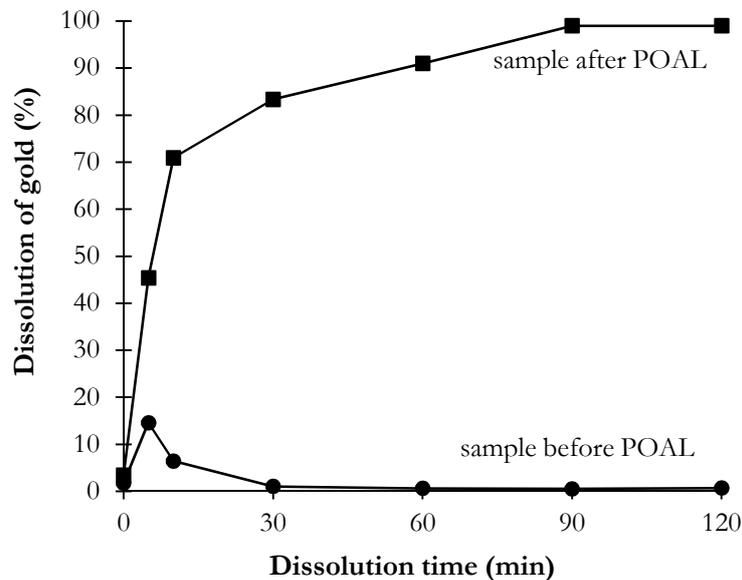


Fig. 2. Gold dissolution from samples before and after POAL in an iodine-iodide solution. 0.3 g/L I₂, 1.8 g/L KI, 1 % pulp density, at 550 rpm and 40 °C.

It suggested that the higher concentrations of base metals (Cu, Al, Fe and Zn) in the WPCBs sample before POAL cause of difficulty in gold leaching from the sample in an iodine-iodide solution, whereas the vast majority of gold (> 99 %) in the WPCBs sample after POAL was dissolved in the iodine-iodide solution (Fig. 2). The results of these experiments provided evidences that the pressure oxidative acid leaching (POAL) is needed to treat WPCBs sample in order to remove base metals especially Cu and Al and to promote the maximizing the efficiency of gold extraction from the sample in the iodine-iodide solution.

3.2. Iodine-Iodide Leaching

The variables affecting the efficiency of the iodine-iodide leaching process; such as iodine/iodide concentration, pulp density, leaching time and leaching temperature were separately investigated in order to optimize gold leaching from the WPCBs sample after POAL in the iodine-iodide solution. Among them, some important variables for gold dissolution are discussed in the following sections.

3.2.1. Effect of Leaching Temperature

Effect of temperature on the efficiency of gold dissolution was studied under the condition which were iodine concentration of 0.3 g/L, KI concentration of 1.8 g/L, pulp density of 1 % with varying leaching times at 550 rpm. As shown in Figure 3, the dissolution efficiency of gold increased drastically with increasing the temperature and reached to 99 % after 90 min leaching at 40 °C and 60 °C, respectively and become constant further. Whereas the efficiency of gold leaching at 20 °C was 67 % at 90 min and increased with the increase in leaching time and then reached 92 % after 240 min leaching. This observation relates to the kinetic reaction control and iodine species generated in the solutions with different temperatures.

3.2.2. Effect of Iodine Concentration and Iodine Consumption

The gold leaching experiments were conducted using different concentrations of iodine ranging from 0.1 g/L to 6 g/L with the iodine/iodide molar ratio of 1:6 when the other variables, which are pulp density, conduct time, leaching temperature and agitation speed, were kept constant. Figure 4 shows the correlation

between concentration of iodine/iodide and iodine consumption with regards to the gold dissolution under the same leaching conditions.

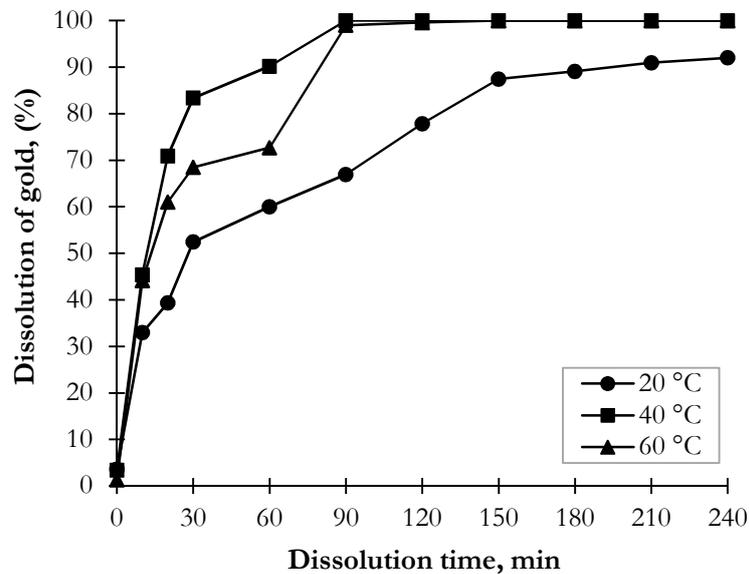


Fig. 3. Dissolution of gold as functions of leaching time and temperature. 0.3 g/L I₂, 1.8 g/L KI (molar ratio of I/KI = 1: 6), 1 % pulp density, 550 rpm.

It can be seen that gold dissolution increased drastically from 20.7 % to 96.4 % with increasing the iodine concentration from 0.1 g/L to 6 g/L while iodine consumption raised from 0.1 g/L to 3.2 g/L (Fig. 4). The iodine concentration of 2 g/L was selected as an optimum condition for further leaching study, because 1.3 g/L (65 %) iodine consumed to 91 % of gold dissolution from the sample. An increase in gold leaching efficiency in the iodine-iodide solution associates with the consumption of iodine and predominant iodine/iodide species such as I⁻, I₂, I₃⁻, IO₃⁻ and IO₄⁻ formed in the solution.

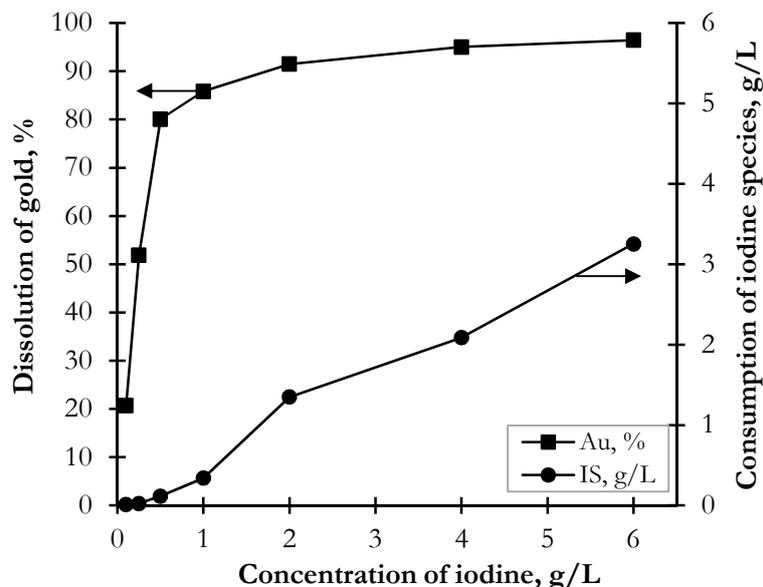


Fig. 4. Dissolution of gold as functions of iodine concentration and iodine consumption. 0.1– 6.0 g/L I₂, 0.6 – 36 g/L KI, 5 % pulp density at 550 rpm and 40 °C for 2 h.

Concentrations of the species such as I⁻, I_{2(aq)}, I₃⁻ and IO₃⁻ in the iodine-iodide solutions were estimated by R. Naumov method using the STABCAL software. The figures 5 and 6 represent the concentrations of

predominant species against standard reduction potential in the solutions containing 0.5 g/L and 2 g/L iodine at 40 °C, respectively. The various concentration profiles of the I_3^- species were observed in the iodine-iodide solutions contained 0.5 g/L and 2 g/L iodine (Figs. 5 & 6).

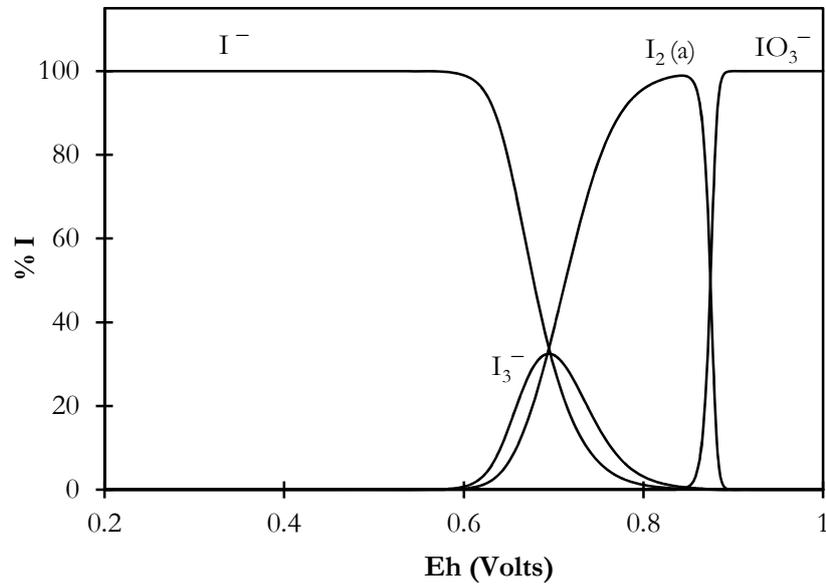


Fig. 5. Concentrations of various species of iodine, 0.5 g/L I_2 , pH 4 at 40 °C, (by STABCAL).

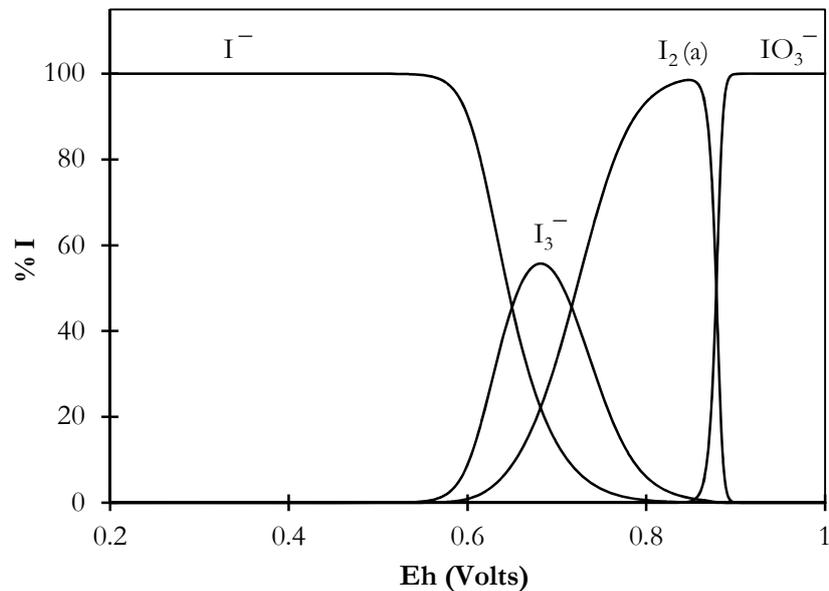


Fig. 6. Concentration profiles of different species of iodine, 2 g/L I_2 , pH 4 at 40 °C, (by STABCAL).

According to Eqs. 1-3 and 7, the different iodine/iodide species especially tri-iodide (I_3^-) is a main oxidant in the iodine-iodide solution for efficient gold dissolution at the temperature.



Our result is consistent with the conclusion of other authors published in the literature [22, 23]. They reported that tri-iodide (I_3^-) is the predominant oxidant in the iodine-iodide solution and the higher concentration of iodide is essential to its formation.

3.2.3. Effect of Leaching Time

The effect of leaching time on the dissolution of gold from the WPCBs sample was studied under the various leaching times changing from 1 to 48 hours while keeping the iodine concentration of 2 g/L, molar ratio of iodine/iodide of 1:6, pulp density of 5 %, agitation speed of 550 rpm at 40 °C. As shown in Fig. 7, gold leaching efficiency increases with increasing the leaching time from 1 to 12 hours and it becomes a constant until 24 hours. The maximum gold dissolution rate reached nearly 99 % when the leaching time was 12 hours.

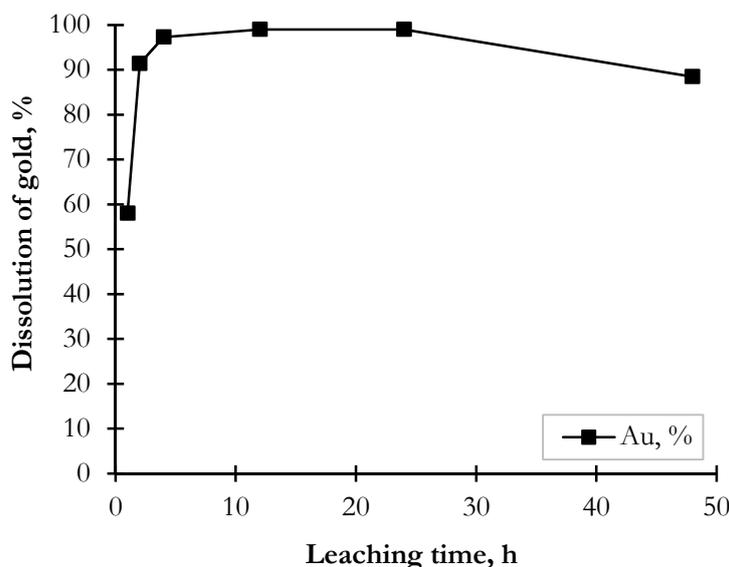


Fig. 7. Dissolution of gold as a function of leaching time.
2 g/L I₂, 12 g/L KI, 5 % pulp density at 550 rpm and 40 °C.

The effects of other variables such as pulp density and leaching temperature were also studied in order to optimize the gold leaching from the WPCBs sample into the iodine-iodide solution. Under the optimized conditions, the vast majority of gold (> 99 %) in the sample was dissolved into the iodine-iodide solution from a high pulp density slurry. It suggested that higher concentrations of iodine and iodide are required to the formation of tri-iodide (I₃⁻) ion which allow to efficient extraction of gold from the high density slurry. There was no obvious difference in the XRD patterns between the 2 samples as before and after iodine-iodide leaching.

3.3. Gold Adsorption onto the Activated Carbon

Adsorption tests were conducted on the pregnant solution resulting from the iodine-iodide leaching by using activated carbon. Different adsorption parameters like iodine/iodide concentration, gold concentration, particle size of activated carbon, carbon dose, pH of pregnant solution, shaking speed and contact time and temperature were investigated in order to optimize the gold adsorption process. For instance, some important parameters which influence on the gold adsorption from the pregnant solution/leach liquor are shown below.

3.3.1. Effect of pH on the Gold Adsorption

The initial pH of the pregnant solution obtained from iodine-iodide leaching under the optimum conditions was pH of 3.2. The effect of pH of the pregnant solution on gold adsorption was studied in broad ranges of pH changes between pH 2 and pH 12 which was adjusted by addition of 1 M HCl and 1 M KOH, respectively. As can be seen from the results shown in Figure 8 (A), the pH changes between pH 2 to pH 10 did minor affect the adsorption of gold from the solution. The maximum adsorption (74 %) of gold took place at the pH of 3.2 which is an initial pH of the solution, while the adsorption of iodine was

0.9 g/L. On the contrary, the variable (pH change) had effect on the adsorption of iodine species that increased with increasing the pH of the solution. The adverse effect on gold adsorption have at the lower and higher pH levels, respectively. Precipitation of gold was occurred when increase the pH of the solution from pH of 10.5. An optimum pH value for gold adsorption from the iodine-iodide solution was selected as the pH of 3.2. The species distribution values for various pH are obtained using STABCAL software and are plotted as a Dist-pH diagram. As shown in Figure 8 (B), the gold molecules precipitate in the high pH range and ionic species such as Au^{3+} exist and dominate in the acidic pH range.

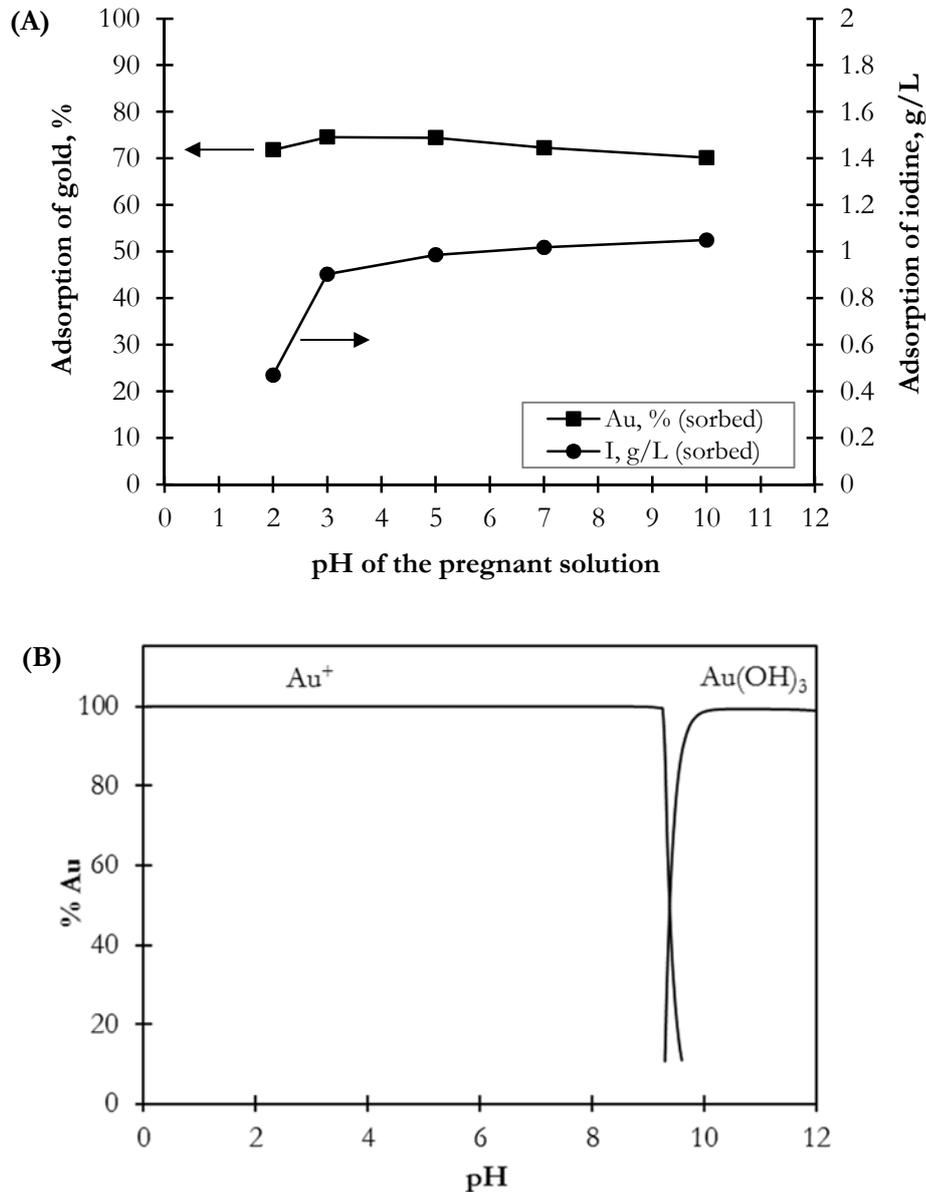


Fig. 8. (A) Adsorption of gold and iodine species on the activated carbon as a function of pH of the pregnant solution, B) Distribution of gold species in the solution as a function of pH; 35.2 mg/L Au, 20 ml pregnant solution, 20 mg activated carbon, < 75 μm particle size, 1.25 g/L I_2 , 120 rpm shaking, 30 min at 20 $^\circ\text{C}$.

3.3.2. Effect of Iodine Concentration on the Gold Adsorption

Pregnant solutions were prepared using different concentrations of iodine ranging from 0.25 g/L to 4 g/L under the optimum leaching conditions determined in previously (Section 3.2). According to the various iodine concentrations used in the leaching process, the concentrations of gold and iodine in the pregnant

solutions are varied from 15 mg/L to 40 mg/L and from 0.2 g/L to 2.4 g/L. The adsorption efficiencies of gold and iodine are determined by ICP-OES and redox titration with sodium thiosulfate and the results estimated are plotted in Fig. 9. The adsorption of gold decreases with increasing the adsorption of iodine onto the activated carbon, as shown in Fig. 9. The results suggested that the both gold and iodine are sorbed onto the carbon simultaneously and the higher concentration of iodine species in the pregnant solution has a negative effect for the gold adsorption on the carbon.

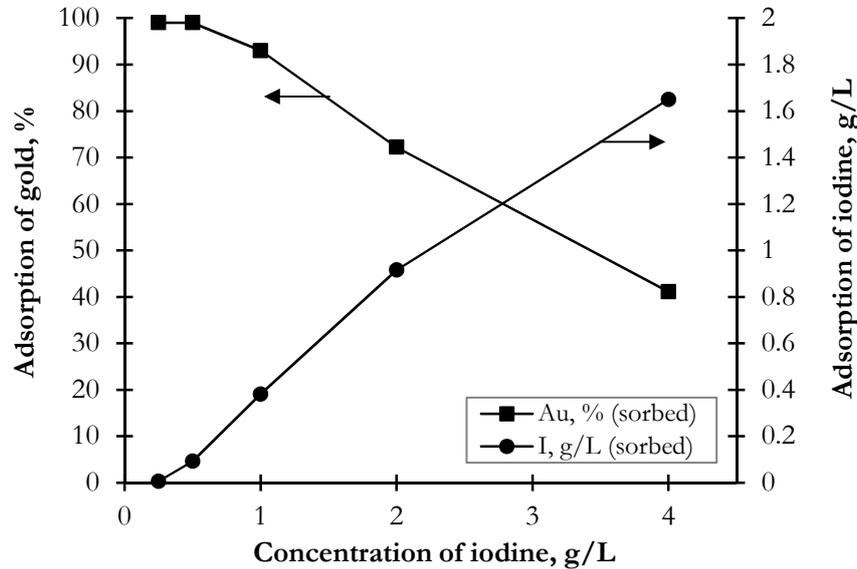


Fig. 9. Adsorption of gold and iodine on the activated carbon as a function of concentration of iodine in the lixiviant; 15 – 40 mg/L Au, 20 ml pregnant solution, 20 mg carbon, < 75 μm particle size, 0.25 - 4 g/L I_2 , pH of 3.2, 120 rpm shaking speed, 30 min at 20 $^\circ\text{C}$.

As mentioned above, adsorption variables such as gold concentration in the solution, particle size of activated carbon, carbon dose, shaking speed, contact time and temperature were investigated for optimization of this process. Under the conditions optimized more than 98 % of gold was adsorbed onto the activated carbon. FTIR measurement revealed 2 peaks at 2199 cm^{-1} and 2032 cm^{-1} which are maybe related to loaded gold-iodide species on the carbon (Fig. 10).

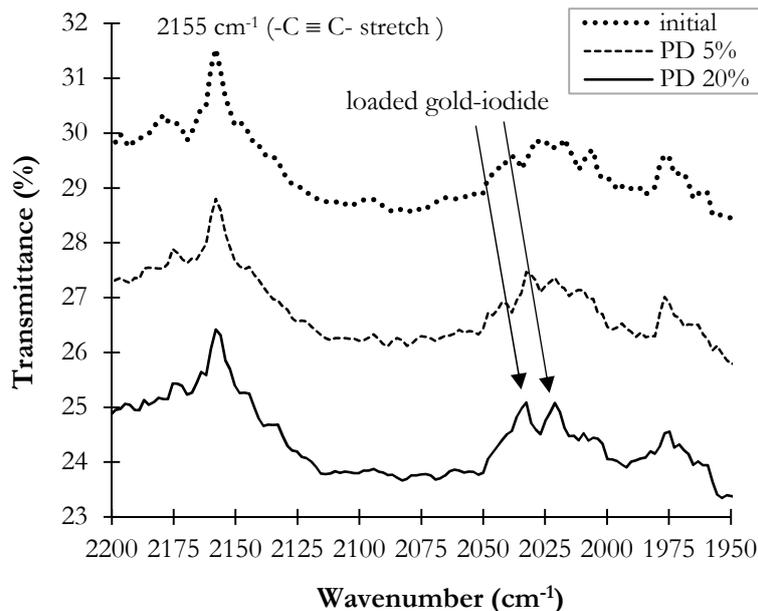
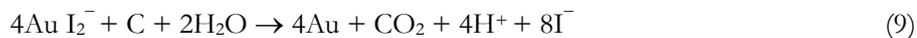
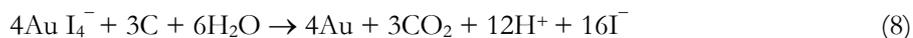


Fig 10. FTIR spectra of the carbon loaded gold and iodine from gold-iodide solutions; Au: 32 - 105 mg/L, pulp density (PD): 5 % and 20 %, particle size of the carbon \approx 75 μm .

SEM-EDS analysis detected major elements such as Au and iodine from the carbon obtained by the adsorption (no figure). These measurements confirmed the sorption of Au and I onto the carbon simultaneously. Mechanism of gold adsorption from leach liquor onto activated carbon can be described by Equations 8 & 9 [24].



4. Conclusions

This work focused on the recovery of gold from the waste printed circuit boards (WPCBs) by using iodine-iodide leaching followed by activated carbon adsorption. Preliminary tests revealed that the higher concentrations of base metals (Cu, Al, Fe Zn), especially Cu in WPCBs showed a negative effect on gold dissolution into the iodine-iodide solution. In order to remove the base metals and upgrade gold content in the residue sample of WPCBs, a pressure oxidative acid leaching (POAL) was conducted in an autoclave using a diluted sulfuric acid solution. Dissolution of Cu, Al, Fe and Zn were 99.3 %, 82.3 %, 61.2 % and 96.4 % respectively under the selected conditions. A batch experiment was conducted to optimize the gold dissolution from the solid residue of WPCBs sample into the iodine-iodide solution using various variables such as iodine/iodide concentration, pulp density, leaching time and leaching temperature. Results indicate that the vast majority (more than 99 %) of gold was dissolved in the solution from the residue under the optimized leaching conditions which were established to be 2 g/L for iodine, 12 g/L for iodide, 550 rpm for stirring at 40 °C for 120 min. Adsorption tests were performed on pregnant solutions resulting from the iodine-iodide leaching using activated carbon. Different adsorption parameters like iodine/iodide concentration, particle size of the carbon, carbon dose, pH, shaking speed and temperature were investigated.

The optimum conditions for the gold adsorption from the pregnant gold-iodide solution were found to be as follows: particle size of carbon, <75 µm; carbon dose, 1 g/L; pH of the pregnant solution, 3.2; shaking speed, 140 rpm; temperature, 20 °C and adsorption time, 30 min. Under these conditions, more than 98 % of gold was adsorbed onto the activated carbon. It was revealed that a higher concentration of iodine can promote the efficiency of gold dissolution in the iodine-iodide solution, in contrast to this phenomenon, excess iodine (iodine/iodide species) in the pregnant solution has a negative effect on the gold adsorption onto the activated carbon. Iodine and gold loaded onto the carbon could be recovered by stripping using NaOH, acids (H₂SO₄, HNO₃, HCl) and aqua regia followed by precipitation using reducing and oxidizing agents, respectively. As a result of this study, nearly 97 % gold can be successfully recovered from the WPCBs as a secondary resource.

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