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Recovery of Au, Ag and Cu from printed circuit board leachate using activated carbon derived from foxtail fruit

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Abstract. Printed circuit boards (PCBs) are the e-waste generated from the end-of-life electronic equipment such as laptops and mobile phone. PCBs contain various metals including precious metals (gold, silver, copper) and detrimental heavy metals as well (arsenic, mercury) [9]. Recycling of e-waste is potentially to be one of the best mechanisms to overcome the human and environmental health threat hence, the valuable metals can be recovered and could avoid the depletion of our ore resources. In this paper, hydrometallurgical process on PCBs was carrying out to recover the precious metals. The PCBs were immersed into aqua regia leaching solution and later the targeted metals were leached out and extracted using activated carbon through adsorption process. The precious metals were then recovered by desorbing the spent activated carbon using hydrochloric acid (HCl) as desorbing agent. In this study, foxtail palm fruit was used to produce activated carbon for metals recovery process. Therefore, the objective of this study was to evaluate the efficiency of the prepared activated carbon derived from foxtail fruit for the recovery of Au, Ag and Cu contain in the PCB leachate. The effect of adsorbent dosage (1, 2, 3, 4, 5g), contact time (20, 40, 60, 80, 100 min) and desorption process of spent activated were investigated. The characterization of the prepared activated carbon was determined using field emission scanning electron microscope (FESEM) whereas the PCBs leachate solution before and after metal recovery process were quantified using flame atomic absorption spectrophotometer (FAAS). The obtained result showed that, the adsorption percentage of Au, Ag and Cu at high adsorbent dosage (5g) with longer contact time (100 min) were 65.51%, 30.30% and 62.51% respectively. However, the attained result for desorption percentage of the metals recovery for Au, Ag and Cu were recorded to be higher at shorter contact time (20 min) as the spent activated carbon could deteriorate at longer contact time with concentrated HCl. The percentage recovery values for 20 minutes desorption process were 99.77% (Au) when 5g of activated carbon was used, whilst 97.41% (Ag) and 98.83% (Cu) were obtained when 2g of activated carbon were applied, respectively. Thus, it can be concluded that the adsorption of Au, Ag and Cu were greater when higher dosage of activated carbon and longer contact time were applied. Meanwhile, shorter contact time were needed to recover the metals. Therefore, this study could be one of the finding in safeguarding our environment by minimizing the e-waste pollution as well as practicing metal recycling in community.

1. Introduction

Electronic devices or gadgets have become a central issue for society as it provides multipurpose use that makes life convenient. For instance, the mobile phone has become one of the essentials as it is a medium for communication whilst a computer that could store information and data systematically [1]. However, the escalating demand for electronic devices would be a reason for the fast growth of e-waste generation and the improper management of e-waste disposal have emerged to be one of the world's issues that could pose potential threats to environmental and human health [1]. According to Brune *et al.,* [3] e-waste has many detrimental heavy metals such as mercury (Hg), lead (Pb), nickel (Ni) and other metals. Exposure to e-waste has shown a relation to adverse effects in environment and human health such as fetal loss, prematurity, congenital malformation and others. Despite that, according to Kaya [9], e-waste contains many valuable metals such as aurum (Au), argentum (Ag) and copper (Cu). Hence it can be considered as both secondary resources and environmental toxicant. Moreover, personal computers or laptops and mobile phones are among abundant e-waste where printed circuit boards (PCBs) play a vital role to support and connect electronic components and thus PCBs are the backbone of the electronic devices [9]. PCBs that weigh about 3% of a whole are recorded to be the most economically attractive portion of waste electrical and electronic equipment (WEEE) as it contains a diverse mixture of metals, non-metals and even some of the toxic substances. Besides, it has been reported that 1 metric ton of waste PCBs consist approximately 80-1500g of Au and 160-210kg of Cu. The PCB comprises a diverse range of metals and precious metals, which hold its own function such as Ag is use as protection against oxidation. Meanwhile, the chip of the circuit boards consists of Au and Ag as the connectors and Al as the capacitors [9]. Large amount of Cu in the circuit boards is used as base metals. Thus, it is beneficial to recycle and recover the valuable metals in the PCBs. The PCB is shown in Figure 1.

Figure 1. Printed Circuit Board (PCB).

Generally, there are several ways of recycling and recovering the precious metals from the PCBs such as hydrometallurgy and pyrometallurgy/incineration. However, previous studies have reported that hydrometallurgy is a promising process for the metals recovery ranging across all concentrations [9]. This process also has been proven by several studies to be advantageous compared to other metals recovery processes as it is environmentally safe where it does not emit any harmful gaseous compound and does not generate particulates matter along the operation [9]. Figure 2 below shows the overall flow process of the hydrometallurgy.

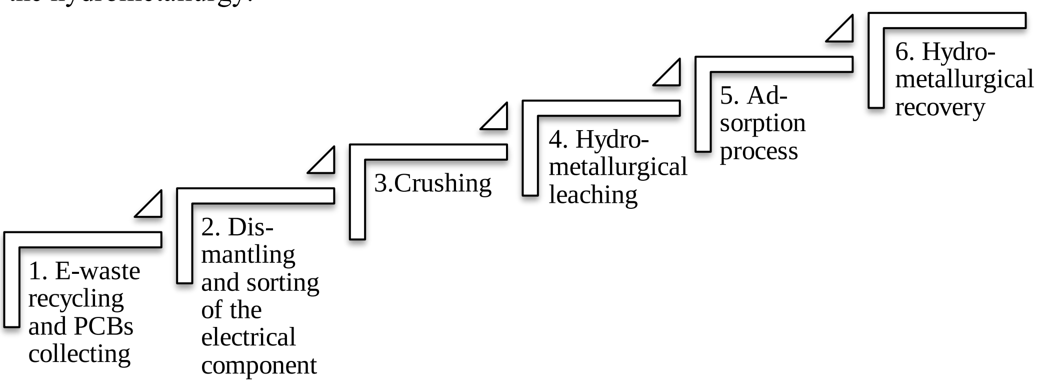

Figure 2. Flow process of hydrometallurgy.

Basically, in hydrometallurgy, the metals from the PCB are leached into solution using a leaching agent. Subsequent to the process, the targeted metals will be recovered and refined via adsorption process [14]. Moreover, the adsorption process using activated carbon has been extensively used for removing metal from polluted solution. The production of activated carbon from agro-waste would be a better option as it is less expensive and environmentally friendly. It has been reported that activated carbon produced by agro-waste has high carbon content and is capable of adsorbing heavy metals from aqueous solution. In this paper, activated carbon has derived from foxtail palm fruit (*Wodyetia bifurcate*) and utilized to adsorb the targeted metals. The fruit is attractive because of the bright red colours with a rounded, symmetrical shape as shown in Figure 3. The tree also is an adaptable, fast-growing plant that would be a great garden decoration [17].

Figure 3. The tree of foxtail palm fruit, *Wodyetia. Bifurcate.*

Foxtail palm fruit has been studied as a biosorbent for the removal of Pb(ll), Fe (ll) and Zn (ll) [19]. Furthermore, in recovery of precious metals through adsorption studies, a desorption process is needed to recover all the metals that have been adsorbs. There are few chemicals that has been listed to act as a desorbing agent such as hydrochloric acid (HCl), sulphuric acid (H_2SO_4), phosphoric acid (H_3PO_4), nitric acid (NaOH), sodium chloride (NaCl) and ultrapure water [13]. However, one of the studies found out that HCl was the efficient desorbing agent with 86% efficiency [13]. Therefore, the adsorption of precious metals from PCBs leachate using activated carbon derived from foxtail palm fruits agro-waste may contribute to turning into valuable product such as activated carbon besides reducing the agro-waste; recycling on the valuable metal such as Au, Ag and Cu that can be recovered and later can be used as secondary sources for the other processes. This study could also promote to improving environmental and human health as it minimizes the e-waste pollution.

2. Materials and Methods

In this study, several processes were conducted to achieve the end result which was to recover the Au, Ag and Cu from PCBs leachate using activated carbon derived from foxtail palm fruit and also to determine the re-usability of the prepared activated carbon. Figure 4 below shows the flow process of the study.

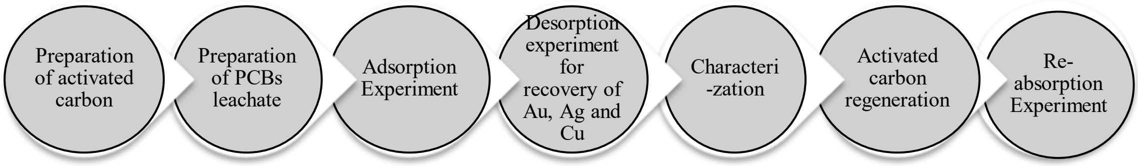

Figure 4. The flow process of the study.

2.1. Preparation of Activated Carbon from Foxtail Palm Fruit, Wodyetia Bifurcate

The foxtail palm fruits were washed with distilled water to remove any impurities and dirt that has been on the surface of the fruits. The cleaned fruits then were dried in the oven at 100ºC overnight and later were cooled down to room temperature. Next, the dried fruits were carbonized at 300ºC in the furnace for two hours and were cooled down to room temperature as well. Figure 5 below shows the preparation process of the raw foxtail fruits until the fruits were carbonized respectively.

Figure 5. The preparation process of the raw foxtail fruits to carbonized fruit.

Subsequent to the process, the carbonaceous char was crushed and grinded using miller blender and were sieved to pass through a 250mm mesh sieve [19]. Next, for the chemical activation process, 40g of the powdered char was soaked and impregnated in 80ml of HNO_3 [11]. The mixture was mixed vigorously until it became paste. In order for the chemical to be fully reacted, the paste then was left in the fume hood overnight. After that, the slurry was placed in a dry crucible and carbonized in the furnace at 500ºC for two and half hours [12]. Finally, the produced activated carbon was washed using distilled water to remove any excess of HNO_3 and pH adjusted until it reached pH 7. The activated carbon was oven dried at 100~150ºC for three hours and kept in a polyethylene bag and stored in a desiccator for further use [11]. Figure 6 below shows the process of the char to activated carbon.

Figure 6. Foxtail fruit char to activated carbon preparation.

2.2. Preparation of Printed Circuit Board (PCB) Leachate
In order for the leaching process to be able to leach faster and thoroughly, the collected PCBs were dismantled, sorted, shredded to the smallest pieces (1~5cm). Then, the samples were leached using a leaching agent which in this study, aqua regia leaching solution were used to leach the sample. 20g of the sample were soaked in 1000ml of aqua regia and left for 24 hours for the leaching process to be fully completed. Finally, the leachate solution was filtered and stored in 2000ml of Duran reagent flask in the dark for further use. The preparation of the PCBs leachate is shown in Figure 7.

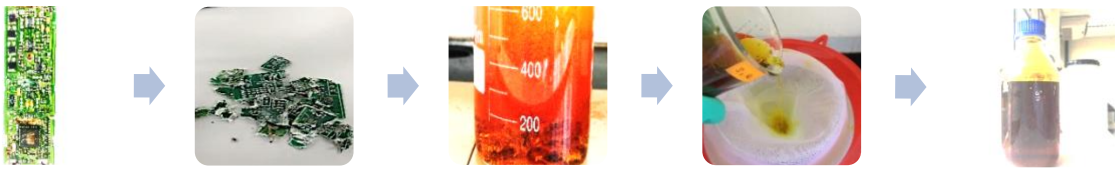

Figure 7. The preparation process of PCB to leachate solution.

Moreover, the aqua regia leaching solution was prepared using 1:3 mixtures of concentrated HNO_3 and HCl. As, aqua regia is extremely corrosive with fuming yellow solution, all procedures were conducted in the fume hood with appropriate personal protective equipment (PPE) such as eye goggles, chemical respirator, nitrile glove, lab coat along with long pants and closed-toe shoes [6]. Figure 8 below shows the aqua regia leaching solution.

Figure 8. Aqua regia leaching solution.

2.3. Adsorption Experiment
Adsorption experiment were conducted using 100ml of PCBs leachate solution in 250ml of Erlenmeyer flask by varying different parameters such as adsorbent dosage (1, 2, 3, 4, 5g) respectively and contact time of 20, 40, 60, 80 and 100min. In each experiment, three repetitions were done and the flask was agitated at 150 rpm to ensure homogeneity and accuracy. After the adsorption experiment were completed, each sample was filtered and later characterized using a flame atomic spectrophotometer (FAAS). The percentage adsorption of Au, Ag and Cu by the activated carbon from foxtail fruits were determined using equation (1) [2].

$$Percentage\ Adsorption = \frac{Ci - Cf}{Ci} \times 100 \qquad (1)$$

Where C_i is the initial metal concentration and C_f is the final reading of the metal concentration in the leachate solution.

2.4. Determination of Percentage of Yield
The percentage of yield (%) of the produced activated carbon from foxtail fruits was determined by the following equation (2) [10].

$$Percentage\ of\ Yield\ = \frac{Wf}{Wi} \times 100 \qquad (2)$$

Where W_f is the final mass of the activated carbon at the end of chemical activation process and W_i is the initial mass of the dry impregnated char.

2.5. Desorption Experiment for Recovery of Au, Ag and Cu
Desorption experiment were conducted by using the spent activated carbon from the previous adsorption experiment where different spent adsorbent dosages (1, 2, 3, 4 and 5g) were soaked in 50ml of HCl desorbing agent and agitated up to 200 rpm for 22 hours with the temperature slightly higher than room temperature which was 33.8ºC as the rate of desorption is higher with higher temperature [4]. The samples then were filtered and also characterized using FAAS. The percentage recovery of Au, Ag and Cu were determined using equation (3) [2].

$$Percentage\ Recovery\ (R\%) = \frac{Ci - Cf}{Ci} \times 100 \qquad (3)$$

Where C_i is the initial reading of the metal concentration of the PCB leachate and C_f is the final reading of the metal after the desorption process.

2.6. Characterization
The prepared activated carbon was characterized using field emission scanning electron microscope (FESEM) in order to observe the surface morphology of the activated carbon and determine its porosity and type of pores available respectively.

2.7. Activated Carbon Regeneration
The regeneration of activated carbon was done for the selective spent sample only. Whereas, the spent samples that achieved the highest recovery rate were selected to be regenerated. In this study, 5 and 2g of the spent activated carbon has recorded the highest percentage recovery (R%). Hence, 5 and 2g of the spent activated carbon were first oven dried at 100ºC overnight prior to use. Next, the spent samples were pyrolysed in a furnace at 750ºC with regeneration duration ranging from 0.5 hours to 3.0 hours. The regenerated activated carbon then was stored in a desiccator for reused [15].

2.8. Re-adsorption Experiment for Re-usability
The regenerated activated carbon from foxtail fruit was tested for re-usability. The experiment was conducted using 100ml of PCBs leachate solution in 250ml Erlenmeyer flask with 5 and 2g adsorbent dosage of regenerated activated carbon. The samples were agitated at 150 rpm for 100 min (the highest percentage adsorption was at longer contact time). Later, the samples were filtered and characterized in FAAS. The percentage absorption was calculated using equation (1) [2].

3. Result and Discussion

3.1. Percentage of Yield
The percentage yield of foxtail palm fruit activated carbon impregnated with a ratio 2:1 of HNO_3 and carbonised at 500ºC was 59.42% which was comparatively high compared to a study on oil palm shell

activated carbon by Kouotou *et al.,* [10] where the percentage yield was 56.47% at 630ºC activation temperature. The modest difference in yield between these palm species could be attributed to the utilisation of a higher temperature for the oil palm shell activation procedure. The microstructure of activated carbon deteriorates as temperature rises [10]. As a result, more volatiles are released [2].

3.2. Characterization

3.2.1. Activated Carbon Surface Morphology. The pore structure of the produced activated carbon was observed by using field emission scanning electron microscope (FESEM). The image captured is shown in Figure 9 below.

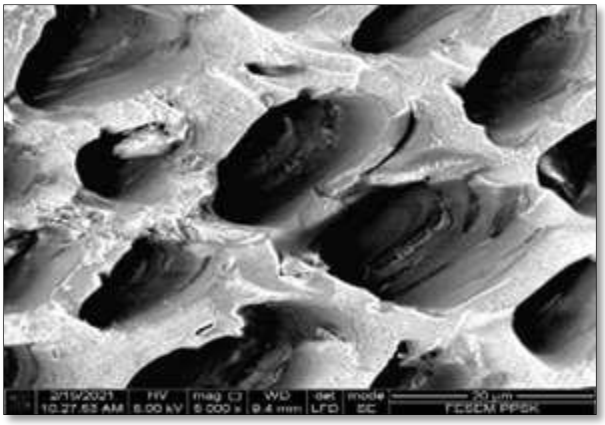

Figure 9. Honeycomb structure of activated carbon using FESEM with 5 x 10^3 magnifications.

From the image, the honeycomb structure was captured existed on the surface of the activated carbon resulted from the evaporation of a chemical reagent (HNO_3) that is used during the carbonization process. The formed structure creates a lot of pores and internal surface area, providing high contact efficiency between the substrate and the flow stream which is crucial for an effective adsorbent [7].

3.3. The Effect of Adsorbent Dosage

The dose of adsorbent was an important parameter to regulate both availability and adsorption sites' accessibility [18]. The adsorption experiment using activated carbon derived from foxtail palm fruits was conducted varying with different adsorbent dosage which was 1, 2, 3, 4 and 5g respectively. The effect of adsorbent dosage on the adsorption of Au, Ag and Cu was presented in Figure 10,11 and 12 below.

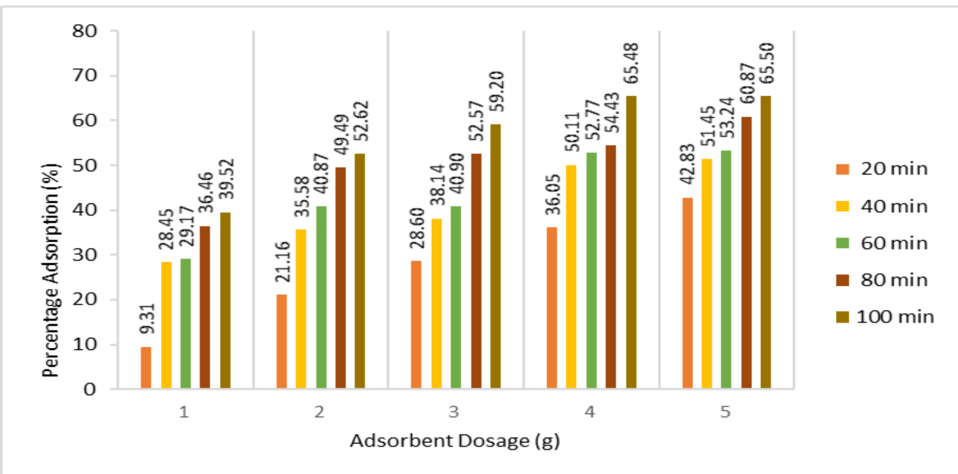

Figure 10. Percentage adsorption of Au with different adsorbent dosages and contact time.

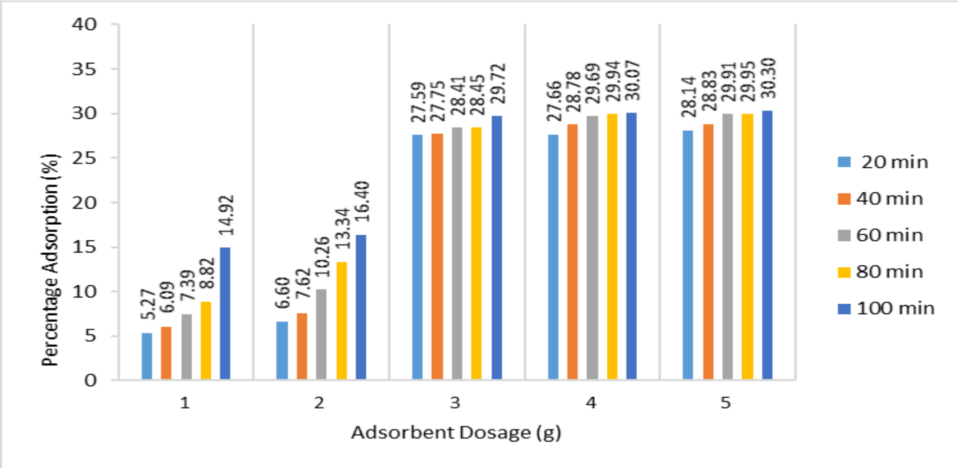

Figure 11. Percentage adsorption of Ag with different adsorbent dosages and contact time.

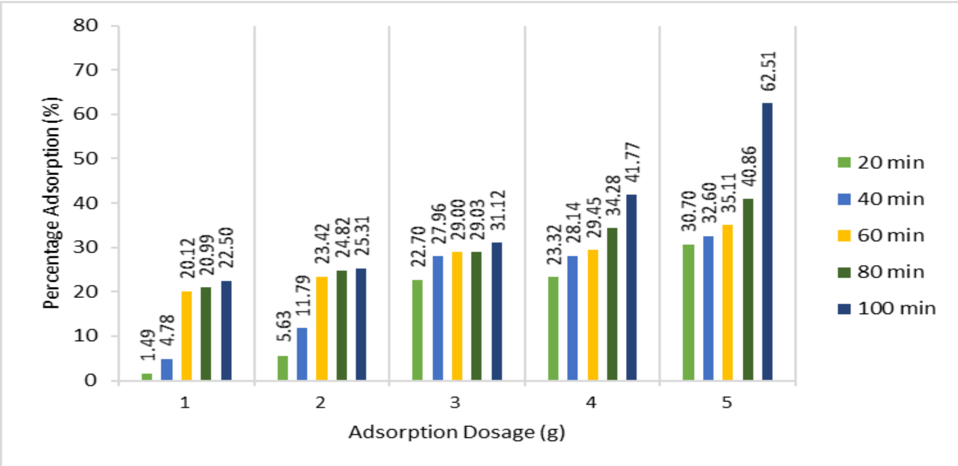

Figure 12. Percentage adsorption of Cu with different adsorbent dosages and contact time.

The adsorption of Au, Ag and Cu metals were recorded to be highest at 5g dose; Au (65.50%), Ag (30.30%) and Cu (62.51%) respectively. Meanwhile, all three metals were recorded to have the lowest adsorption percentage at the lowest adsorbent dosage which was 1g dose; Au (9.31%), Ag (5.27%) and Cu (1.49%). The obtained result shows an inclining reading with the increasing of adsorbent dosage. This is due to more surfaces and functional groups on the adsorbent with which metals can interact. These functional groups were crucial in the establishment of Van der Waals bonding because they were responsible for binding metals to the adsorbent during the adsorption process [21]. Thus, provided more opportunities for adsorption to occur as there was less rivalry amongst metals for binding sites. Hence higher dosage of adsorbent will increase adsorption.

3.4. The Effect of Contact Time

Experimental studies were carried out with differentiating contact time (20, 40, 60, 80 and 100 min) on the Au, Ag and Cu metals adsorption. Figure 10,11 and 12 show an increasing trend of the graph with the increase of contact time at the highest adsorbent dosage (5g). Hence, Figure 13 below has been plotted to distinguish the result.

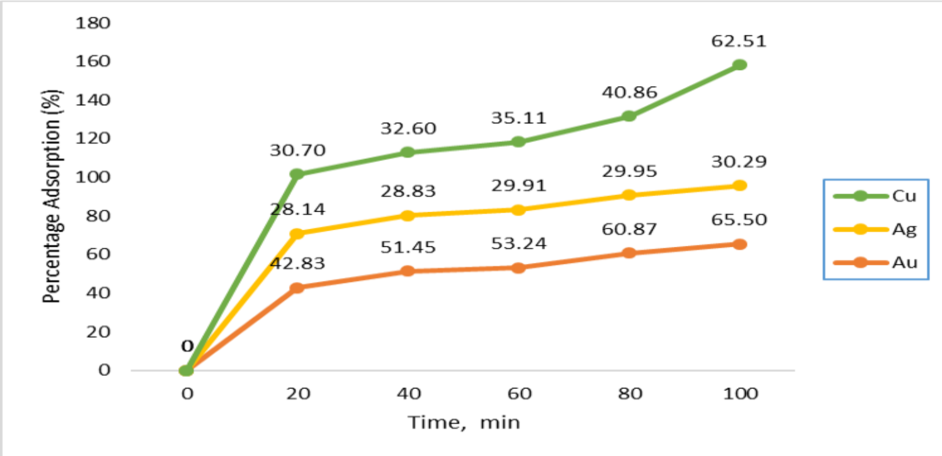

Figure 13. The effect of contact time on 5g dose of Au, Ag and Cu metal adsorption.

The data clearly shows that the agitation time necessary for maximum Au, Ag and Cu metal uptake by the activated carbon was at 100 min. This was because longer contact time would give the binding sites of the activated carbon to bind and interact with the targeted metals and also strengthen the bond with each other [20].

3.5. Desorption Studies for Au, Ag and Cu Recovery

To achieve practical recovery for Au, Ag and Cu metals, it is necessary to desorb the adsorbate. The adsorbate was desorbed using HCl desorbing agent in accordance with the result obtained from a study that has been conducted by Kulkarni *et al.,* [13] on the activated carbon regeneration and recovery; in comparison to other desorbing agents such as sulphuric acid (H_2SO_4), phosphoric acid (H_3PO_4), sodium chloride (NaCl) and ultrapure water, HCl was observed to be the most effective desorbing agent with 86% efficiency. The result of the desorption process for the recovery of Au, Ag and Cu are presented in Table 1, 2 and 3.

Table 1. Recovery (%) of Au metal from foxtail fruit spent activated carbon using HCl.

Contact time (min)	Adsorbent dosage (g)				
	1	2	3	4	5
20	70.12	80.00	93.09	98.39	99.77
40	71.39	89.02	94.34	98.90	99.22
60	94.73	94.78	96.75	99.11	98.11
80	95.70	92.83	96.81	99.49	97.02
100	95.80	98.37	96.83	99.55	97.38

Table 2. Recovery (%) of Ag metal from foxtail fruit spent activated carbon using HCl.

Contact time (min)	Adsorbent dosage (g)				
	1	2	3	4	5
20	96.66	97.41	96.12	95.17	91.69
40	96.70	96.34	95.83	95.12	89.77
60	97.05	96.09	95.34	94.99	89.43
80	97.10	96.01	95.09	94.95	89.18
100	97.32	96.01	93.05	94.85	88.57

Table 3. Recovery (%) of Ag metal from foxtail fruit spent activated carbon using HCl.

Contact time (min)	Adsorbent dosage (g)				
	1	2	3	4	5
20	97.37	98.83	93.20	92.05	96.02
40	97.61	98.81	93.16	91.73	95.41
60	97.70	98.77	91.44	91.66	95.01
80	97.80	98.68	91.28	91.42	94.95
100	97.96	98.58	91.13	90.85	93.67

The attained result showed that Au achieved an increasing data of recovery percentage along with the increasing amount of adsorbent dosage used and up to a certain contact time. The highest reading for the recovery of Au (99.77%) was recorded to be at 5g dose with 20 min of contact time and perceived a decreasing reading afterwards. Ag and Cu was observed to have an increasing recovery percentage with the increasing contact time at 1g dose. Both Ag and Cu metals obtained the highest recovery percentage at 2g dose with 20 min of contact time; Ag (97.41) and Cu (98.83). However, the reading for Ag and Cu were discovered declining with higher adsorbent dosage used and longer contact time. This is due to the fact that longer contact time with concentrated HCl desorbing agent can cause the spent activated carbon to deteriorate [13]. Moreover, the reason for the recovery percentage to be decreasing is probably because the metal ions were likely bound to the pores of activated carbon and through strong interaction, reducing desorption potential over time [20]. Nevertheless, the acquired recovery percentage for Au was escalating with the increasing of contact time up to 4g adsorbent dosage. This was resulted as HCl is a by-product of hydrogen (H^+) and chlorine (Cl^-). The chlorine present in the HCl solution oxidizes the Au metal efficiently [5].

3.6. Regeneration and Reusability of Adsorbent

Regeneration process of the adsorbate was carried out to determine the capability of the produced activated carbon from foxtail fruit to regenerate and to be re-used. Regeneration of activated carbon also could prevent the cause of secondary sources of pollution as the waste activated carbon is normally incinerated or discharged [15]. Thermal regeneration is a promising method for decomposing and converting organic matter into biochar, bio-oil, and permanent gases in the absence of oxygen at temperatures between 150 and 700ºC [15]. In this study, the spent activated carbon that obtained the highest recovery percentage from the previous desorption process were chosen to be regenerated; Au (5g dose and 20 min of contact time), Ag and Cu (2g dose and 20 min of contact time). The spent activated carbon was pyrolysed in the furnace at 750ºC and was tested for its effectiveness to re-adsorp Au, Ag and Cu from PCBs leachate. In order for the regenerated activated carbon to achieve maximum adsorption, both of these different adsorbent dosages (2g and 5g) were evaluated for a total of 100 minutes of contact time. The loss rate of pyrolysis was calculated and demonstrated in Table 2.

Table 4. The loss rate of regenerated activated carbon.

Adsorbent Dosage (g)	Mass of activated carbon after dried in oven (before regeneration) (g)	Mass of activated carbon after pyrolysis (after regeneration) (g)	Loss rate of pyrolysis (%) ($\frac{\boldsymbol{m1-m2}}{\boldsymbol{m1}} \times \mathbf{100}$)
2	1.69	0.83	51.18
5	4.62	2.48	46.26

The thermal regeneration of activated carbon for 2g and 5g adsorbent dosages was observed to have a mass loss with up to 50% loss rate of the pyrolysis; 2g (51.18 %) and 5g (46.46%). This was resulted as an addition to the pyrolyzed organic adsorbate residues, the spent activated carbon is partly gasified or over-burned [8]. Furthermore, the experiment was further carried on to the adsorption process and the result is shown in Figure 14 below.

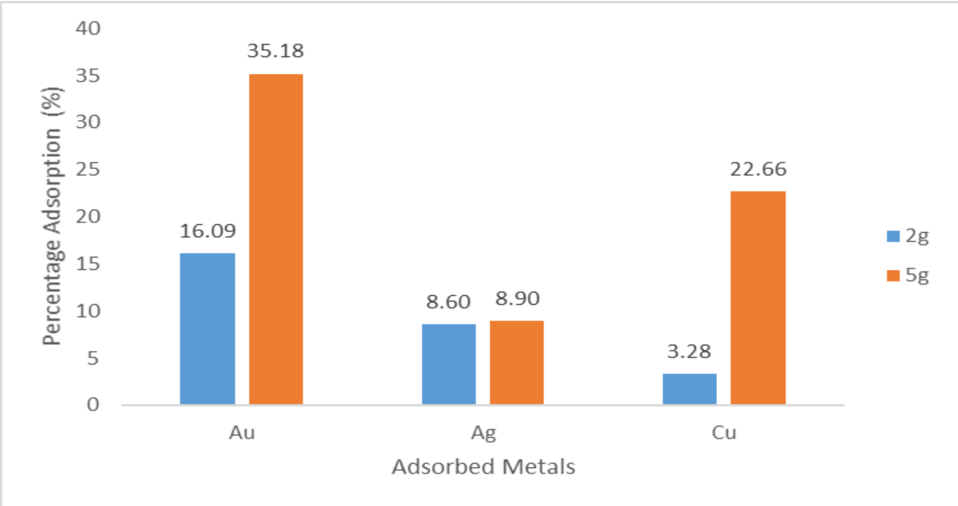

Figure 14. Percentage adsorption of Au, Ag and Cu after 100min of contact time with different adsorbent dosage.

It is clearly shown that the percentage adsorption of Au, Ag and Cu was recorded to be higher at 5g adsorbent dosage; Au (35.18%), Ag (8.90%) and Cu (22.66%) compared to 2g dose used; Au (16.09%), Ag (8.60%) and Cu (3.28%) respectively. Nevertheless, this percentage adsorption was relatively low and this could be due to the mass loss of the adsorbent after pyrolysis. Initially, 5g and 2g of the adsorbent dosage had a mass loss for about 2g~ and 1g~ dose, resulting in the reduction of the binding site for the adsorption process [8]. A study that was conducted by Li *et al.,* [15] on the regeneration of waste powder activated carbon through pyrolysis determined that the mass loss in the activated carbon during pyrolysis was divided by three phases; 1) moisture desorption where all the water in the sample easily stripped past the boiling point, 2) thermal desorption and 3) carbonization of solid pyrolysis residue. However, from the attained result, it was found out that spent activated carbon could be regenerated and later to be re-used.

4. Conclusion

This study showed that the hydrometallurgical leaching process could leach the printed circuit boards into solution, allowing the concentration of Au, Ag and Cu to be evaluated using FAAS before and after the adsorption process. Moreover, in this study, the foxtail palm fruits used to produce activated carbon were locally and abundantly available agricultural waste products. Therefore, foxtail fruit activated carbon could be a cost-effective alternative to commercially available activated carbon in the recovery of Au, Ag, and Cu metals. Furthermore, the results clearly demonstrate that higher adsorption percentage of Au, Ag and Cu can be achieved with a greater adsorbent dosage and a longer contact time. Besides, through this study, the pyrolysis regeneration method was discovered to be capable of regenerating spent activated carbon and capable of being re-used. Hence, the secondary pollution by the spent activated carbon could be reduced.

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