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ACTIVATED CARBON FROM FOXTAIL PALM FRUIT *Wodyetia bifurcata* AS POTENTIAL HEAVY METAL ADSORBENT FROM PRINTED CIRCUIT BOARD LEACHATE

(Karbon Teraktif daripada Kelapa Sawit Ekor Musang, *Wodyetia bifurcata* sebagai Potensi Penjerap Logam Berat daripada Air Larut Lesap Papan Litar Bercetak)

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Abstract

The production of activated carbon from the fruit of the foxtail palm, *Wodyetia bifurcata*, was tested as an effective adsorbent for heavy metals (Au, Ag, and Cu). The activated carbon was chemically impregnated in a 2:1 ratio of concentrated nitric acid (HNO_3) to foxtail fruit char, followed by carbonization in a furnace at 500 °C for two and a half hours. In this study, a field emission scanning electron microscope (FESEM) and BET-N_2 adsorption were used to investigate the physical and chemical properties of the prepared activated carbon. In this work, parameters such as the surface area, total pore volume, average pore diameter, physical surface morphology of the activated carbon and its adsorption capacity in adsorbing Au, Ag, and Cu from the leachate of printed circuit boards were investigated. The results showed that the Brunauer, Emmett, and Teller (BET) surface area of foxtail palm fruit activated carbon was 0.680 m^2/g, while the average pore diameter and total pore volume were 7.2366 nm and 4.005 cm^3/g, respectively, indicating that the prepared activated carbon had a remarkable mesopore surface area with the formation of a honeycomb structure. The pore size of the activated carbon was reduced after the adsorption process, indicating that the metals Au, Ag, and Cu were adsorbed on the activated carbon. The highest adsorption percentages of Au, Ag, and Cu were obtained with a high amount of prepared adsorbent (5 g) with a high contact time (100 min) of 65.50%, 30.29%, and 62.51%, respectively. In contrast, the percentage adsorption of Au, Ag, and Cu by commercially available activated carbon (5 g, 100 min), was 70.82%, 46.30%, and 32.64%, respectively. In addition, the adsorption rate was better at a temperature of 25 °C than at 75 °C. As a result of this study, it was found that activated carbon from the fruit of the foxtail palm can be used as a potential adsorbent for the adsorption of heavy metals from the leachate of printed circuit boards.

Keywords: activated carbon, foxtail palm fruit, heavy metal, printed circuit board, leachate

Abstrak

Penghasilan karbon teraktif daripada buah sawit ekor musang, *Wodyetia bifurcata* telah diuji sebagai penjerap yang berkesan untuk penjerapan logam berat (Au, Ag, dan Cu). Karbon teraktif telah diresapi secara kimia menggunakan nisbah 2:1 asid nitrik pekat (HNO_3) kepada arang buah sawit ekor musang, diikuti dengan dua setengah jam pengkarbonan dalam relau pada suhu 500ºC. Dalam kajian ini, mikroskop elektron pengimbasan pelepasan medan (FESEM) dan penjerapan BET-N_2 telah digunakan untuk mengkaji sifat fizikal dan kimia karbon teraktif yang dihasilkan. Dalam kerja ini, parameter seperti luas permukaan, jumlah isipadu liang, purata diameter liang, morfologi fizikal permukaan karbon teraktif dan kapasiti penjerapannya dalam menjerap Au, Ag, dan

Cu daripada air larut lesap papan litar bercetak telah disiasat. Keputusan menunjukkan bahawa luas permukaan Brunauer, Emmett, dan Teller (BET) karbon teraktif buah sawit ekor musang ialah 0.680 m^2/g, manakala purata diameter liang dan jumlah isipadu liang ialah masing-masing 7.2366 nm dan 4.005 cm^3/g, menunjukkan bahawa karbon teraktif yang dihasilkan mempunyai kawasan permukaan mesoliang yang luar biasa dengan pembentukan struktur sarang lebah. Saiz liang karbon teraktif telah mengecil selepas proses penjerapan, menunjukkan bahawa logam Au, Ag, dan Cu telah terjerap pada karbon teraktif. Peratusan penjerapan tertinggi bagi Au, Ag, dan Cu dicapai pada amaun tinggi (5 g) penjerap yang dihasilkan dengan masa sentuh yang tinggi (100 min) iaitu masing-masing pada 65.50%, 30.29%, dan 62.51%. Sebaliknya, peratusan penjerapan Au, Ag, dan Cu oleh karbon teraktif komersial tersedia (5 g, 100 min) ialah masing-masing 70.82%, 46.30% dan 32.64%. Tambahan pula, kadar penjerapan menunjukkan prestasi yang baik pada suhu 25°C berbanding 75°C. Hasil daripada kajian ini, didapati bahawa karbon teraktif daripada buah kelapa sawit ekor musang boleh digunakan sebagai penjerap berpotensi untuk menjerap logam berat daripada air larut lesap papan litar bercetak.

Kata kunci: karbon teraktif, buah sawit ekor musang, logam berat, papan litar bercetak, air larut lesap

Introduction

Activated carbon is a porous carbonaceous material that is used as an adsorbent due to its properties such as an extremely large surface area per unit volume, enormous adsorption capacity, various porosity structures and a high degree of surface reactivity and can therefore adsorb a large number of molecules in its pores [1]. Therefore, the use of activated carbon has proven to be a powerful platform for a variety of applications, including adsorption, water treatment, removal of pollutants and dyes in wastewater, and odour reduction [1]. However, the high cost of commercial activated carbon prevents its use in most cases. Recognizing this economic disadvantage, numerous researchers have worked to develop low-cost alternatives to produce activated carbon from a variety of carbonaceous and mineral precursors, including a wide range of agricultural wastes [2-4], wood [5], lignite [6], fruit stones [7-8], nutshells [9-10], fly ash [11], minerals [12], and scrap tires [13]. Physical and chemical activation are the two most common methods for producing activated carbon. In physical activation, the material is pyrolyzed in the furnace at high temperatures between 400 ºC and 500 ºC [1]. In chemical activation, the starting material is impregnated with chemical activating agents such as potassium hydroxide (KOH), phosphoric acid (H_3PO_4), and nitric acid (HNO_3), which influence the development and formation of a porous structure in the material [1].

Several studies have documented that the highest adsorption of metal ions was obtained from precursors of palm species activated with the activating agent HNO_3 [14-15]. In the present study, the fruit of the foxtail palm was used as a precursor material to produce activated carbon. The fruit of the foxtail palm, also known as *Wodyetia bifurcata*, is a species of palm tree in the Arecaceae family that is popularly planted as a landscape plant in Malaysia and around the world due to its exceptional beauty, as the ripe fruits are bright red in colour and have a peculiar shape resembling a foxtail [16]. The tree is also an adaptable, fast-growing tree that would make a great garden ornamental. However, foxtail fruits have no specific use and are always left to rot. Previous studies have reported that agricultural wastes from palm species, such as oil palm trunks, have been investigated and successfully used as effective activated carbon to remove chromium [17] and as biosorbents to recover Au(III) from $AuCl_3$ solutions [18]. Therefore, the present study aims to utilize the fruit of the foxtail palm as a more valuable product such as activated carbon by using HNO_3 as a chemical activating agent.

The produced activated carbon was then tested for its potential to remove heavy metals from the leachate of electronic waste (e-waste) through the adsorption process. In [19], it was reported that 53.6 million metric tonnes (Mt) of e-waste globally in 2019, showing an increasing trend of up to 21% in just five years. Meanwhile, it was estimated that Malaysia generated 364 kilotons (kt) of e-waste in 2019, which corresponds to an average of 11.1 kg per capita [19]. This makes e-waste the fastest growing household waste stream in the world, mainly due to the high consumption of electrical and electronic equipment, short life cycles, and limited repair alternatives [20]. Personal computers, laptops, and cell phones are examples of e-waste where the printed circuit boards (PCBs), which consist of most of the metals embedded in the circuit board, play an important role in holding and connecting electronic components [20]. This waste is toxic to the environment but contains significant amounts of valuable metals such as gold (Au) and silver (Ag), which makes it recyclable [21]. E-waste, on the other hand, is considered an environmental pollutant because it contains hazardous metals such as copper (Cu), which can pose a health risk to humans and the environment [21]. Therefore, researchers around the world are experimenting with different technologies to treat e-waste such as oxidation, reduction, precipitation, membrane filtration, ion exchange, and adsorption [20]. Nevertheless, adsorption is very successful and cost-effective in all approaches [20]. Therefore, the aim of the present study is to evaluate the effectiveness of activated carbon produced from the fruit of foxtail palm in the removal of Ag, Au, and Cu metal from PCB leachates by adsorption and to compare its removal performance with that of commercial activated carbon to recognize its potential as an alternative adsorbent.

Materials and Methods

Foxtail palm fruit activated carbon preparation

The sample of foxtail palm fruit was collected from Jeli, Kelantan, Malaysia and stored in a zipper bag. The sample was first washed in distilled water to remove any impurities or dirt that had accumulated on its surface. The sample was then dried overnight in an oven at 100 °C and later cooled at room temperature. The char was then crushed, ground with a blender, and sieved through a 250 mm mesh sieve [22]. Subsequently, 40 g of the pulverized char was soaked in 80 mL of HNO_3 and impregnated for the chemical activation process [23]. The mixture was vigorously mixed until it became a paste. The paste was then kept in the fume hood overnight to allow the chemical to react completely. The slurry was then placed in a dry crucible and carbonized in a 500 ºC furnace for two and a half hours [23]. Finally, the prepared activated carbon was washed with distilled water to remove excess HNO_3, and the pH of the washed solution was pH 7. The activated carbon was dried in the oven at 100~150 ºC for three hours, stored in a polyethylene bag, and stored in a desiccator for further use [24]. Figure 1 shows the prepared foxtail palm fruit activated carbon.

Printed circuit board (PCB) leachate preparation

In this study, Au, Ag, and Cu metal were extracted from the leaching process of printed circuit boards (PCBs). The printed circuit boards were initially collected from an e-waste recycling centre in Kelantan (Laksana Cergas Sdn Bhd) located in the industrial area in Pengkalan Chepa, Kelantan, Malaysia. The collected PCBs were disassembled, sorted, and crushed into small pieces (1~5 cm) for faster leaching [25]. The samples were then leached with a leaching agent. In this case, an aqua regia solution was used to leach the samples [25]. The aqua regia leach was prepared by combining concentrated HNO_3 and HCl in a 1:3 ratio [25]. Since aqua regia is highly corrosive and gives off a fuming yellow solution, all processes were carried out in a fume hood, wearing appropriate personal protective equipment (PPE), such as safety goggles, chemical respirator, nitrile glove, lab coat, long pants, and closed shoes [26]. Subsequently, 20 g of the sample was soaked in 1000 mL of aqua regia solution for 24 hours to ensure that the leaching process was complete [25]. The process of leaching PCBs with aqua regia solution is shown in Figure 2. Finally, the leaching solution was filtered and stored in a 2000 ml Duran reagent flask in the dark for further use.

Figure 1. Foxtail palm fruit activated carbon

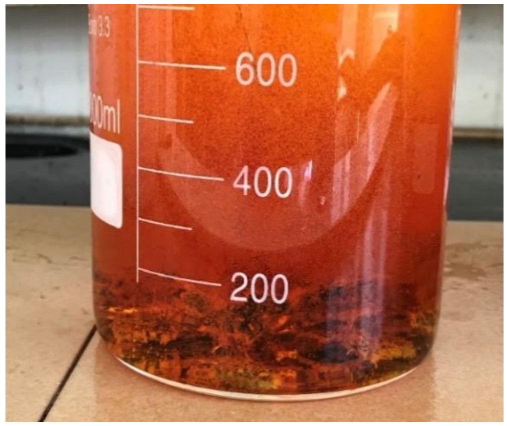

Figure 2. The leaching process of PCBs using aqua regia solution

Removal of Au, Ag, and Cu metal from PCBs leachate using foxtail palm fruit activated carbon through the adsorption process

Adsorption experiments were conducted to evaluate the ability of activated carbon from foxtail palm fruits to remove Au, Ag, and Cu metal from PCB leachate. First, 100 ml of PCB leachate solution was poured into a 250 ml Erlenmeyer flask and shaken with different amounts of adsorbent (1, 2, 3, 4, 5 g) and contact times of 20, 40, 60, 80, and 100 min. To ensure homogeneity, the flask was shaken in each experiment at 150 rpm in an IKA KS 4000i shaker machine [27]. The percentage adsorption of Au, Ag, and Cu by the foxtail palm fruit activated carbon was determined using Equation 1 [27].

$$\text{Percentage Adsorption} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

where C_i is the initial metal concentration and C_f is the final reading of the metal concentration in the leachate solution. The study on the effect of temperature was selected for only 5 g of activated carbon produced at a contact time of 100 min, varying the temperature at 25°C, 50°C, and 75°C.

Removal of Au, Ag, and Cu metal from PCBs leachate using commercial activated carbon through the adsorption process

This study was conducted in the same way as the experiment with foxtail palm fruit activated carbon, but the adsorbent used was commercial activated carbon with the fixed amount of adsorbent (5 g).

Field emission scanning electron microscopy (FESEM) analysis

A Quanta 450 FEG Field Emission Scanning Electron Microscope (Quanta FEG 450; FEI Company, Hillsboro, Oregon, USA) was used to analyse the morphology of foxtail palm fruit activated carbon before and after the adsorption process of Au, Ag, and Cu metals. Prior to the analysis, the samples were mounted on a FESEM holder with double-sided electrically conducting carbon-adhesive tabs to protect the specimens' surfaces from the electron beam [28]. Subsequently the samples were coated with a thin layer of silver (Ag) using a Leica sputter coating instrument (Leica EM SCD005). Coating the samples with a conductive metal layer inhibits specimen charging and thereby improving imaging quality [29]. To achieve a clear image of the surface morphology of activated carbon, images were captured at a magnification level of 5000x before and after the adsorption process.

Brunauer, Emmett, and Teller (BET) Surface Area Analysis

Nitrogen adsorption-desorption isotherms were determined using Quantachrome Autosorb iQ3 surface analysis (Autosorb-iQ3-MP/Kr; Quantachrome Instruments, Brynton Beach, FL, USA). The isotherms were used to calculate the specific surface area of the activated carbon using the Brunauer-Emmett Teller (BET) technique. Prior to analysis, all samples were degassed in a vacuum at 60 °C for 12 hours at 77 K.

Flame atomic absorption spectrometer (FAAS) analysis

The Au, Ag, and Cu metal contained in the PCB leachates were quantified by FAAS (Perkin-Elmer (PE) 3300) and controlled by a computer autosampler (PEAS 60). Prior to analysis, the PCB leachate solution need to be diluted. The PCB leachate was first filtered using a Whatman GD/X 25 mm syringe filter, 0.45 m PTFE, to ensure that the solution was free of precipitation. Five dilution series were performed to make the PCB leachate less concentrated. The diluted PCB leachate solution was then characterized using a flame atomic absorption spectrometer (FAAS).

Results and Discussion

Surface area and pore size distribution

The porous structure of the activated carbon was characterized by the nitrogen (N_2) adsorption isotherm data at 77 K. The BET surface area (S_B), average pore diameter (D_P) and total pore volume (V_t) of the prepared activated carbon are shown in Table 1. The results obtained show that the BET surface area of the activated carbon from foxtail palm fruits was 0.680 m^2/g, while the average pore diameter and total pore volume were 7.2366 nm and 4.005 cm^3/g, respectively. According to the IUPAC classification, the pores of adsorbents are divided into three types: Micropores (pore diameter < 2 nm), mesopores (2-50 nm), and macropores (pore diameters > 50 nm) [30]. This indicates that the prepared activated carbon had a remarkable mesoporous surface. In addition, the curve of N_2 adsorption/desorption isotherms of the prepared activated carbon chemically activated with HNO_3 is shown in Figure 3. It shows the characterization of a type IV isotherm associated with a mixture of microporous and mesoporous materials [14]. A study conducted by [14] on the effect of HNO_3 treatment on activated carbon from oil palm shells showed a combination of type I (without HNO_3) and IV (with HNO_3) isotherms, suggesting that the porous structure of activated carbon treated with HNO_3 provides a better surface area [14]. Thus, the result indicates that the prepared activated carbon impregnated with HNO_3 could have a highly microporous structure.

Table 1. Surface area, average pore diameter and total pore volume of the prepared activated carbon

BET Surface Area, S_B (m^2/g)	Average Pore Diameter, D_P (nm)	Total Pore Volume, V_t (cm^3/g)
0.680	7.2366	4.005

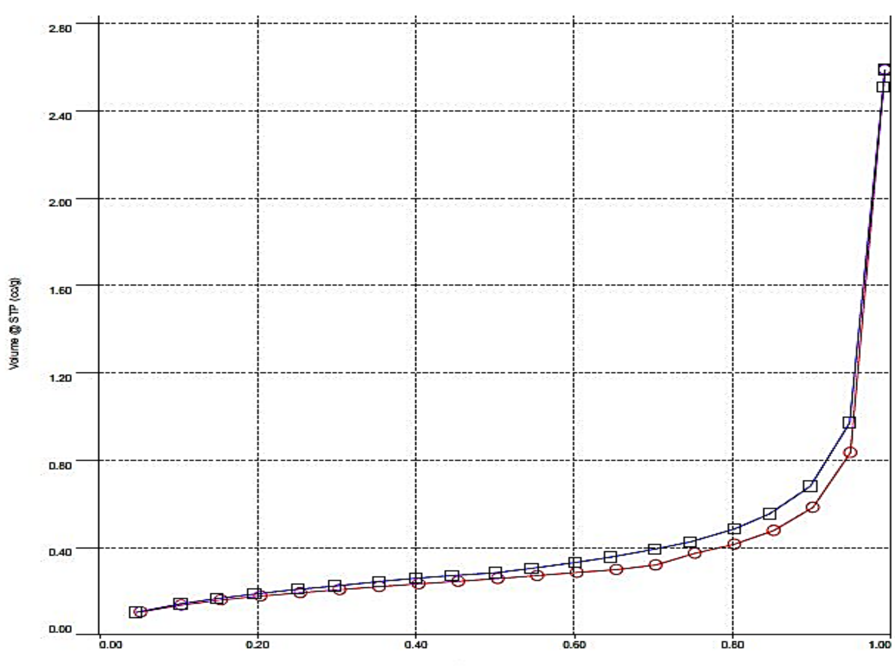

Figure 3. Adsorption/desorption isotherms of HNO_3-activated carbon

Heavy metal content in the PCB leachate

The initial metal concentration of Au, Ag, and Cu from PCB and after the adsorption process using 5 g of prepared activated carbon with a contact time of 100 min is shown in Table 2. The correlation coefficient R^2 for the three metals was 0.999 and the wavelengths for the detection of Au, Ag, and Cu were 242.80 nm, 328.07 nm, and 324.75 nm, respectively.

Surface morphology of the foxtail palm fruit activated carbon

Figure 4(a) shows the surface morphology of activated carbon from the fruit of the foxtail palm at 5000x magnification observed with a field emission scanning electron microscope (FESEM). The result showed that the honeycomb structure on the surface of the activated carbon was formed by the evaporation of a chemical reagent (HNO_3) used during the carbonization process [31]. The formed structure generates many pores and a large internal surface area that ensures high contact efficiency between the substrate and the flow stream, which is important for efficient adsorption [31].

The surface morphology of foxtail palm fruit activated carbon after 100 min adsorption process showed that the honeycomb structure of the activated carbon was maintained even after the adsorption process of Au, Ag, and Cu metal. However, it was found that the pore size of the activated carbon was reduced as the pore surface was cavities distributed after the adsorption process, showing that the Au, Ag, and Cu metal were adsorbed on the activated carbon [31]. Obviously, the pore sizes of the activated carbon were smaller at longer contact times, as can be seen in Figure 4(b). This interesting result could be because the highest adsorption percentage was achieved at a high adsorbent dosage (5 g) with a longer contact time (100 min), as shown in the adsorption test study in the following section, and this was also confirmed by other study [17].

Table 2. Concentration of Au, Ag and Cu in PCB leachate

Heavy metal	**Concentration (mg/L)**	
	Initial	**Final (After Adsorption Process using 5 g of Produced Activated Carbon, 100 min of Contact Time)**
Au	3.467 ± 0.084	1.196 ± 0.0169
Ag	16.230 ± 0.162	11.314 ± 0.167
Cu	14403.330 ± 0.345	5399.808 ± 0.994

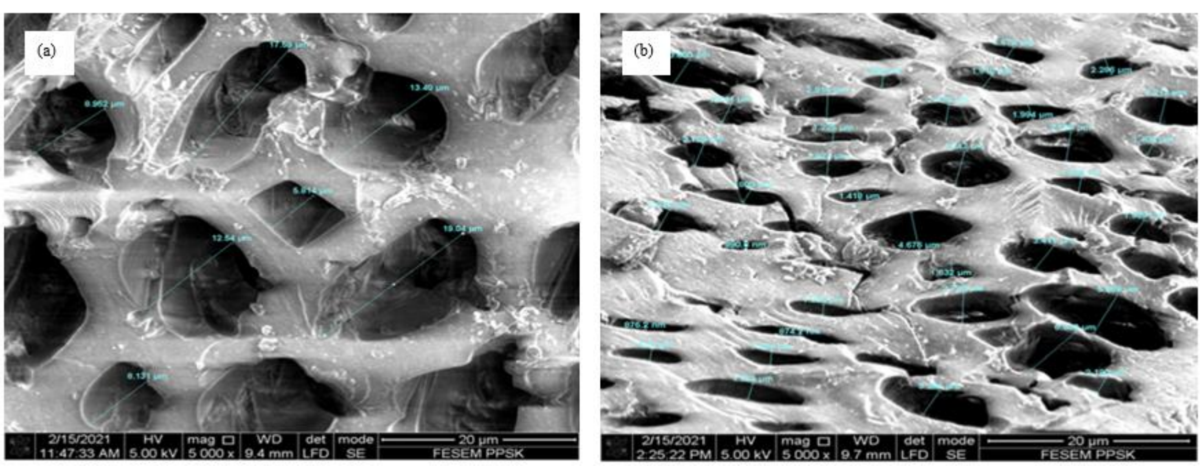

Figure 4. FESEM micrographs of the 5 g of foxtail palm fruit activated carbon (a) before and (b) after 100 mins of the adsorption process of Au, Ag, and Cu at 5000x magnification

Adsorption of Au, Ag and Cu by prepared activated carbon

An adsorption test was conducted to evaluate the effectiveness of the prepared activated carbon (foxtail palm fruit) in adsorbing Au, Ag, and Cu metals from PCB leachate. In the experimental studies, the amount of adsorbent (1, 2, 3, 4, 5 g) and the contact times (20, 40, 60, 80, 100 min) were varied. The adsorption of Au, Ag, and Cu metal was highest (65.50%, 30.29% and 62.51%, respectively) at a high adsorbent dosage (5 g) and longer contact times (100 min), as shown in Figure 5. At the lowest adsorbent dosage of 1 g and shorter contact time of 20 min, all three metals had the lowest adsorption percentages: Au (9.31%), Ag (5.27%), and Cu (1.49%). At high adsorbent levels, more surface areas and functional groups were introduced on the adsorbent for metals to interact with. These functional groups were important for the formation of Van der Waals bonds as they are responsible for the binding of metals to the adsorbent during the adsorption process [32, 33]. As a result, there were more opportunities for adsorption as the metals competed less for the binding sites. Thus, a larger amount of adsorbent increases adsorption and a longer contact time would allow the binding sites of the activated carbon to bind and interact with the desired metals and strengthen the bond between them [33, 34].

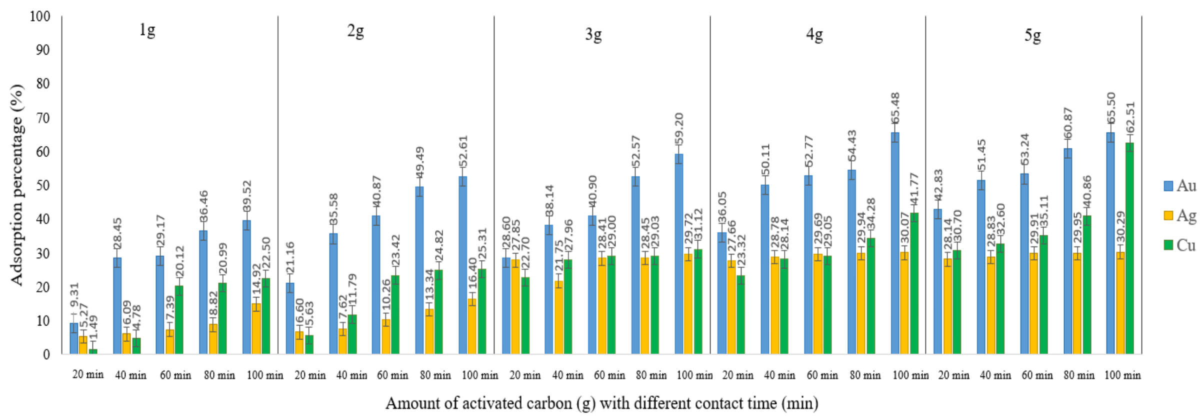

Figure 5. The adsorption percentages of Au, Ag, and Cu metal with various amounts of prepared activated carbon and contact time

Adsorption of Au, Ag and Cu by commercial activated carbon

The present study was conducted to evaluate the performance of commercially available activated carbon in the adsorption of Au, Ag, and Cu metals from PCB leachates. An amount of 5 g of commercial activated carbon was used, as this amount showed the highest adsorption percentage in a previous experiment. The commercial activated carbon showed the lowest trend of adsorption percentage at 20 and 40 min (Figure 6) compared to the prepared activated carbon (Figure 5). However, when Au was removed, the adsorption percentage increased to 71.88%, and 72.34% at 60 and 80 min, respectively. Nevertheless, the adsorption percentage for Au decreased to 70.82% when the contact time was increased to 100 min. A similar trend was observed for Ag removal. At 100 min of contact time, the adsorption percentage of Au (70.82%) and Ag (46.30%) were slightly higher when using commercial activated carbon than with prepared activated carbon (Au: 65.50%, Ag: 30.29%). The results showed only small differences (5-16%) suggesting that the adsorption performance of the prepared activated carbon is nearly comparable to the commercial one. Moreover, the percentage adsorption of Cu by commercially available activated carbon was not as impressive compared to the prepared activated carbon, with values of 29.36%, 30.13%, 30.57%, 30.82%, and 32.64% for 20, 40, 60, 80, and 100 minutes of contact time, respectively. The recent study [35] also came to a similar conclusion, where the peanut shell adsorbent was more effective in adsorbing Pb(II), Cu(II), and Cd(II) than commercial activated carbon, although its specific surface area was 532 m^2/g compared to 3.6 m^2/g (peanut shell).

The effect of temperature

The temperature effect is one of the most important parameters for investigating the performance of the prepared activated carbon in the adsorption of heavy metals. The experiment was carried out with 5 g of prepared activated carbon at a contact time of 100 min. The temperatures tested were 25 °C, 50 °C, and 75 °C. As can be seen in Figure 7, the higher the temperature, the lower the adsorption percentage achieved. This is due to the higher kinetic energy achieved at higher temperatures, which causes the intermolecular interaction between the heavy metal and the surface of the activated carbon to decrease. This result is similar to a previous study [36] on the removal of Pb, and Cd.

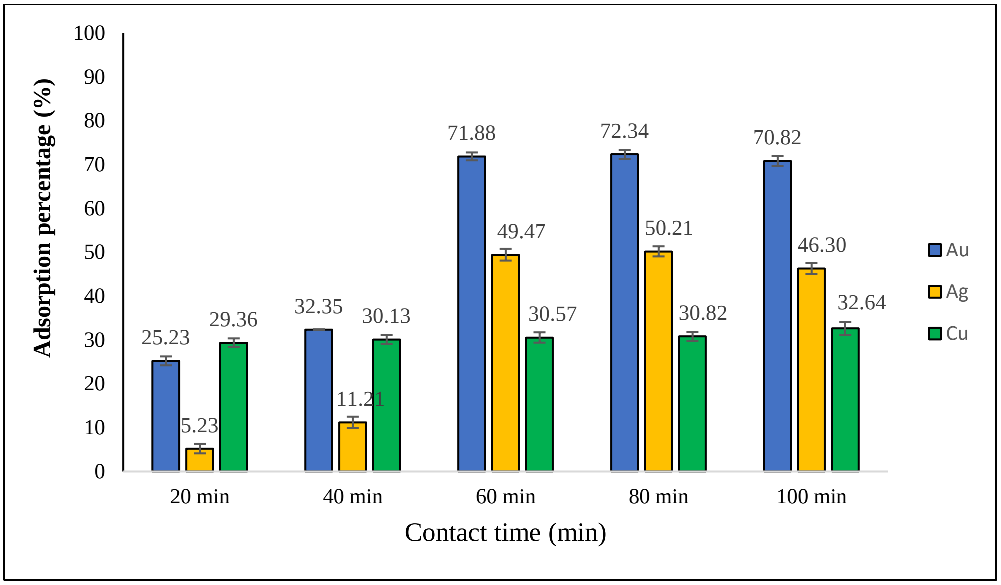

Figure 6. The adsorption percentage of Au, Ag, and Cu metal by 5 g of commercial activated carbon for various contact times

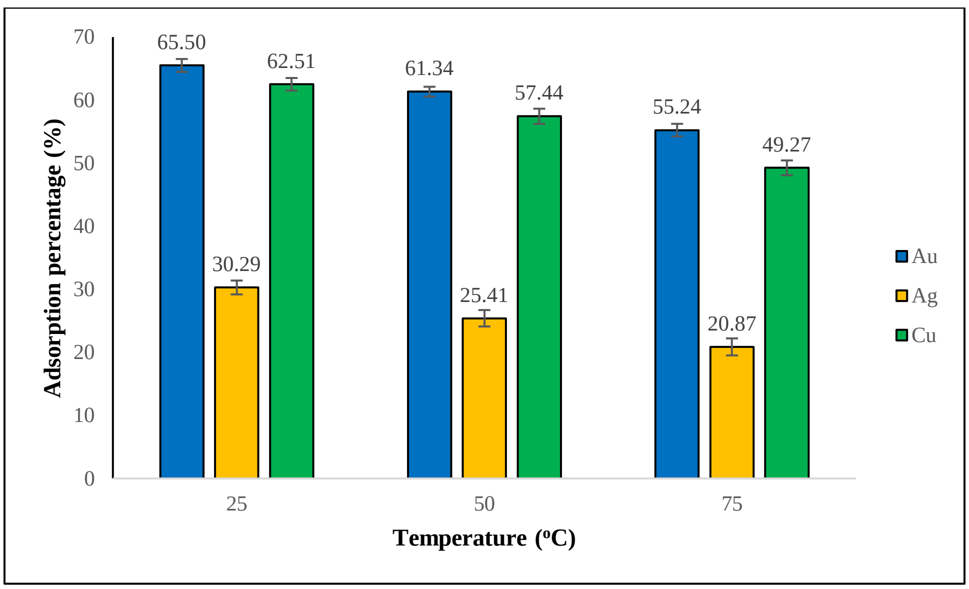

Figure 7. The adsorption percentage of Au, Ag, and Cu metal by 5 g of prepared activated carbon for various temperatures

Conclusions

The present study shows that activated carbon obtained from the fruit of the foxtail palm is a promising adsorbent with distinct properties. Activation of the prepared activated carbon impregnated with nitric acid resulted in mesoporous activated carbon with an average pore diameter of 7.2366 nm. The FESEM micrograph indicated the existence of the honeycomb pore structure and the pores on the surface of the activated carbon after the adsorption process were full of cavities, indicating that the metals Au, Ag, and Cu were adsorbed on the prepared activated carbon. In addition, the result showed that the high adsorption capacity of Au, Ag, and Cu metal was achieved at a longer contact time (100 min) with a larger amount (5 g) of the adsorbent used. Furthermore, it can be said that the prepared activated carbon achieved very similar adsorption performance to the commercial activated carbon. However, the effect of temperature showed a lower percentage adsorption due to the weakening of the intermolecular interaction between the adsorbate and the surface of the prepared adsorbent.

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