Chemically modified palm kernel shell biochar for the removal of heavy metals from aqueous solution

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Abstract. Heavy metals eradication from water is a complicated subject, therefore a viable, resilient, and green technology is imperative. Heavy metal removal can be accomplished through easy access, economical, and efficient sorbents derived from agricultural waste. In the current study, palm kernel shell (PKS) waste was converted into biochar (PKSC) via pyrolysis. Chemical modification was performed on PKSC via acid-base treatment to refine its adsorption properties. Batch experiments were conducted to study the efficiency of PKSC and acid-base treated PKSC (MPKSC) for removal of Cr(IV), Ni(II) and Cu(II). The surface area was increased from 112.934 m²/g to 149.670 m²/g by acid-based treatment. Batch adsorption study showed that the MPKSC afforded high removal efficiency for Cu (99.29%), Ni (96.77%) and Cr (42.97%). The Cr(IV) and Ni(II) adsorption by PKSC, as well as Cr(IV), Ni(II) and Cu(II) adsorption by MPKSC were best represented by Freundlich isotherm. However, Cu(II) adsorption by PKSC can be explained by using Langmuir isotherm. All studied heavy metals fitted the pseudo-second-order kinetic.

1. Introduction
The growth in industrialization and urbanization has led to various environmental issues, including pollution due to heavy metals. Owing to its toxicity to humans, wildlife, and plants, heavy metal contamination is one of the environmental and health issues. Strong metal is poisonous and continues to absorb and induce disease in organisms [1]. Heavy metals are known as inorganic waste that is not biodegradable and persists in the water supply. Electroplating, electronics, batteries, and metal treatment/fabrication are among the main sources of these toxic heavy metals. Electroplating industries generates vast amounts of metal-rich effluents that discharge high concentrations of many hazardous substances, like cyanides, cleaning agents, degreasing solvents, grease, fat, poisonous heavy metals, in particular chromium, nickel, and copper, and surpass the permitted limits [2].

Effective removal of heavy metals from water is crucial for the preservation of drinkable water source. Many techniques have been employed to eliminate or reduce the concentration of heavy metals in wastewater. Adsorption is recognized as one of the best heavy metal removal techniques owing to its functional coherence, low cost, and high performance, when a suitable adsorbent is employed [3], [4]. At the same time, biochar has been applied for many environmental uses, including as adsorbent for heavy metal removal from wastewater. Biochar, a porous carbon-rich material with a large surface area is a product of pyrolysis or carbonization. Many mechanisms are linked to the efficient removal of heavy metals by biochar, including electrostatic interactions, absorption, ion exchange, complexation, and chemisorption [5], [6].
Palm kernel shell (PKS) is an agricultural waste abundantly available in Malaysia, and several applications as adsorbent have been reported earlier. A dried, raw PKS was studied for adsorption of Cr(VI), Pb(II), Cd(II) and Zn(II) [7], and PKS activated carbon for the adsorption of Cr(IV), Cu(II), Ni(II), Pb(II), Fe(II) and Zn(II) [8]. Besides, polyethyleneimine impregnated PKS activated carbon was studied for the removal of Cd(II) and Pb(II) [9], and PKS activated carbon derived from microwave pyrolysis was applied for the removal of Ni(II), Cu(II) and Cr(IV) [10]. In this work, acid-base treated PKS biochar was prepared and applied as an adsorbent for removal of Cr (IV), Ni (II), and Cu (II) from aqueous solution. The adsorption behavior was then studied using Langmuir and Freundlich isotherms.

2. Methodology

2.1. Preparation of adsorbent

Raw PKS was carbonized in a rotary kiln reactor (800 °C, 10 min) to afford PKS biochar. The PKS biochar was soaked in 20% NaOH (2 h), oven-dried (105 °C, 24 h), and mixed with HCl (5 M, 5 mL). The treated PKS biochar was then washed with deionized water to pH 4.5, oven-dried (105 °C, 24 h), and ground to 0.1-1 mm particle size [10]. The raw PKS, PKS biochar (PKSC) and acid-base treated PKS biochar (MPKSC) is shown in Figure 1.

![Figure 1. (a) Raw PKS, (b) PKSC, and (c) MPKSC.](image)

2.2. Characterization of adsorbent

Proximate analyses were carried out based on ASTM D-3173, ASTM D 3174, and ASTM D 3175 to assess the moisture content (MC), ash content (AC), and volatile matter content (VMC). The fixed carbon content (FCC) was calculated from MC, AC and VMC values by difference [10]. The elemental analysis was conducted using CHN Elemental Analyzer (Model: Flash EA1112 Series). The Brunauer, Emmet, and Teller (BET) surface area and pore volume of samples were analyzed using Surface Area Analyzer (Quantachrome® ASiQwin TM). Sample was heated (200 °C, 8 h) under vacuum condition. The nitrogen gas was used as inflow gas. The surface area and pore volume of the biochar sample were determined by measuring the adsorption isotherm of the sample towards the nitrogen gas. The functional groups present in the sample were determined using a Fourier Transform Infrared (FTIR) spectrometer (Model: Perkin Elmer Spectrum GX) at wave number range 400 to 4000 cm⁻¹.

2.3. Preparation of heavy metal solution

Three heavy metals were used in batch adsorption study, namely Cr(VI), Cu(II), and Ni(II). Heavy metal solutions were prepared from 1000 ppm of K₂Cr₂O₇, CuSO₄·(5H₂O) and NiCl₂·6H₂O, respectively. The pH of heavy metal solution was adjusted using 0.1 M NaOH and 0.1 M HCl to pH 7 [11].

2.4. Batch Adsorption Study
Batch adsorption study for Cr(VI), Cu(II), and Ni(II) was carried out using PKS, PKS biochar (PKSC) and acid-base treated PKS biochar (MPKSC). Adsorbent sample (0.5 g) was added into heavy metal solution (25 mL), and the mixture was shaken at room temperature (200 rpm, 1 h) [10]. The mixture was then filtered before heavy metal analysis using Atomic Adsorption Spectroscopy (AAS) (Model: Thermo Specific iCE 3500). The effect of the initial heavy metal solution concentration on the removal efficiency was studied at 5, 10, 15, 20, 30, 50, and 100 ppm concentrations. The effect of contact time on the removal efficiency was studied at 30, 60, 120, 150, 180, 210, and 240 min. The concentration of the solutions (25 mL) was maintained at 30 ppm, adsorbent mass (0.5 g), and mixing speed (200 rpm).

3. Results and Discussion

3.1. Characterization of adsorbent

3.1.1. Proximate Analysis. The results of the proximate analysis are shown in Table 1. Raw PKS had slightly higher moisture content compared to PKSC and MPKSC. High temperature carbonization has reduced the moisture content [12]. The carbonization and acid-base treatment reduced the volatile matter content of the sample, resulting in lower volatile matter content of MPKSC (60.88%) compared to PKS and PKCS. In this analysis, the volatile matter of MPKSC is comparable to previous study on PKS activated carbon [14]. Carbonization allows cellulose and hemicellulose to be decomposed and released as volatile matter, resulting in the formation of microporous structures on adsorbent [13].

| Proximate Analysis       | Raw PKS (%) | PKSC (%)  | MPKSC (%) |
|--------------------------|-------------|-----------|-----------|
| Moisture content (%)     | 9.00±1.41   | 7.25±0.35 | 6.50±0.71 |
| Volatile matter (%)      | 74.79±11.58 | 71.50±7.62| 60.88±7.09|
| Ash content (%)          | 10.00±6.36  | 9.75±1.06 | 4.50±0.00 |
| Fixed carbon (%)         | 6.21±4.51   | 10.25±6.20| 28.12±7.80|

Results also showed that the ash content was decreased after carbonization, and was further reduced after acid-base treatment. A low ash value adsorbent implies a strong adsorbent feature [13]. High ash content indicates the presence of inorganic compounds that hinder the evolution of the surface region by micropores blockage [10]. The MPKSC has 4.50% ash content, which is in agreement with ash content for PKS activated carbon in previous studies [10], [16]. The fixed carbon content is the remaining volume of carbon contained in the biomass after the moisture content and volatile matter have been removed entirely. The fixed carbon content was 6.21% in raw PKS, 10.25% in PKSC, and 28.12% in MPKSC, which is lower compared to PKS activated carbon in previous work [15].

3.1.2. Ultimate analysis. The organic fractions of PKS, PKSC, and MPKSC are described in Table 2. In raw PKS, the high ash and moisture content lead to low carbon content.

| Component | Carbon | Hydrogen | Nitrogen | Sulphur | Oxygen | C/N | C/H |
|-----------|--------|----------|----------|---------|--------|-----|-----|
| Raw PKS (%) | 23.43  | 13.90    | 1.90     | 0.34    | 49.43  | 26.91| 8.66|
| PKSC (%)   | 49.16  | 8.75     | 2.85     | 0.14    | 37.13  | 32.56| 24.38|
| MPKSC (%)  | 51.13  | 9.41     | 2.93     | 0.28    | 36.25  | 30.44| 21.80|
The carbon content of PKSC and MPKSC was observed to be relatively higher compared to raw PKS. The large difference between the raw PKS and the PKSC and MPKSC was due to the carbonization process that increased the carbon content of the sample. The oxygen content percentage was reduced from raw PKSC (49.43%), PKSC (37.13%) to MPKSC (36.25%) due to oxygen emission during carbonization process [17]. Both PKSC (49.16%) and MPKSC (51.13%) showed a lower carbon value compared to previous studies (77-85%) [15]. The hydrogen content in PKSC and MPKSC were lower than previous studies (1.10-4.10%), while the nitrogen content in PKSC and MPKSC was comparable to previous studies [15].

### 3.1.3. Functional group analysis

The FTIR spectra of raw PKS, PKSC, MPKSC is shown in Figure 2. The C-H stretching between the 2925-2850 cm⁻¹ zone in raw PKS and PKSC revealed the presence of alkanes[19]. However, C-H stretching was not found in MPKSC samples. This could be attributed to treatment reactions with acid-base that eliminated the C-H stretching group [18]. C=O stretching at 1750-1650 cm⁻¹ appeared only in raw PKS samples, which is attributed to ketones, aldehyde, and carboxylic acid groups. The presence of alkene group was shown by peaks of C=C stretching between 2990-3000 cm⁻¹ [20] in raw PKS and MPKSC. The peaks at 1470-1350 cm⁻¹ revealed the presence of the C-H in raw PKS, which disappeared in PKSC and MPKSC.

![Figure 2. FT-IR spectrum of raw PKS, PKSC, and MPKSC](image)

### 3.1.4. BET surface area and pore volume

After carbonization and chemical initiation, the surface area and overall pore volume contribute to the removal efficiency of heavy metals [21]. Table-3 shows that the surface area of PKSC and MPKSC were higher than raw PKS. It can be concluded that acid-base treatment on biochar generally provide better surface area and pore structures in modified samples.

|          | Surface area (m²/g) | Total pore volume (cm³/g) |
|----------|---------------------|----------------------------|
| PKS      | 1.200               | n.d.                       |
| PKSC     | 112.934             | 0.09911                    |
| MPKSC    | 149.670             | 0.09917                    |
Various activation processes yield different pore volumes and surface areas. Steam activation process was reported to yield activated carbon with surface area of 1011-1050 m²/g and total pore volume of 0.45-0.60 cm³/g [22], [23]. Chemical activation process afforded surface area between 192.6-953 m²/g and total pore volume of 0.17-0.43 cm³/g [8], [10], [24].

3.2. Effect of initial concentration on heavy metal removal efficiency

Figure 3 shows the plot of heavy metals removal efficiency by PKSC at different solution initial concentrations. Only a small increase in removal efficiency was observed between 5 ppm (93.2%) and 10 ppm (98.2%) of Cu(II), where equilibrium is reached. At concentration higher than 10 ppm no notable change in Cu(II) removal efficiency by PKSC. This is plausibly due to the binding sites on adsorbent were exhausted and no longer effectively available [10]. The removal efficiency of Ni(II) decreased from 20 ppm (98.97%) to 30 ppm (66.87%). This may be attributed to the saturation of the active sites on adsorbent surface [25]. On the other hand, Cr(IV) showed a rapid improvement in the efficiency of removal from 38.9% at 30 ppm to 87.80% at 50 ppm.

![Figure 3](image_url)  
**Figure 3**: Removal efficiency of Cr(IV), Cu(II) and Ni(II) by PKSC at different heavy metal initial concentrations (Adsorption condition: solution pH 7.00, contact time 60 min, adsorbent dosage 0.5 g/25 mL, and mixing speed 200 rpm).

The relationship between initial concentration and removal efficiency of Cr(IV), Cu(II) and Ni(II) by MPKSC is shown in Figure 4. In general, MPKSC showed higher heavy metal removal efficiency compared to PKSC. High removal efficiency of Cu(II) was achieved even at low concentration (5 ppm, 93.5%), and reached maximum removal (99.3%) and equilibrium at 20 ppm. At 5 ppm, satisfactory removal efficiency (83.1%) was demonstrated for Ni(II), before reaching maximum removal efficiency (96.8%) at 20 ppm. However, Ni(II) removal efficiency by MPKSC dropped at initial concentrations higher than 20 ppm, plausibly due to rapid saturation of adsorbent active sites [10]. The Cr(IV) removal efficiency increased rapidly from 5 ppm (42.4%) to 50 ppm (89.2%), but showed lower removal efficiency compared to Cu(II) and Ni(II).
Figure 4. Removal efficiency of Cr(IV), Cu(II) and Ni(II) by MPKSC at different heavy metal initial concentrations (Adsorption condition: solution pH 7.00, contact time 60 min, adsorbent dosage 0.5 g/25 mL, and mixing speed 200 rpm).

3.3. Effect of contact time on heavy metal removal efficiency

The removal efficiency of Cr(IV), Cu(II) and Ni(II) by PKSC is illustrated in Figure 5. Removal efficiency of Cu(II) by PKSC reached maximum (98.1%) at 150 min contact time. Meanwhile, maximum removal efficiency of Ni(II) was 97.1% achieved at 60 min contact time, before a slight decrease in removal efficiency to reach equilibrium at 150 min. However, Cr(IV) showed low removal throughout the studied contact time. The results is in agreement with results shown in Figure 3, whereby only <40% Cr(IV) was removed by PKSC at 60 min contact time and 30 ppm initial concentration.

Figure 5. Removal efficiency of Cr(IV), Cu(II) and Ni(II) by PKSC at different contact time (Adsorption condition: solution pH 7.00, initial concentration 30 ppm, adsorbent dosage 0.5 g/25 mL, and mixing speed 200 rpm).
Figure 6 shows the removal efficiency of Cr(IV), Cu(II) and Ni(II) by MPKSC at different contact time. Low removal efficiency of Cr(IV) (36.8–41.0%) was observed even with prolonged contact time up to 210 min. Results also indicated that maximum removal efficiency for Cu(II) was 97.3% at 120 min, while maximum removal of Ni(II) was 96.7%, achieved at 210 min.

![Figure 6](image_url)

**Figure 6.** Removal efficiency of Cr(IV), Cu(II) and Ni(II) by MPKSC at different contact time (Adsorption condition: solution pH 7.00, initial concentration 30 ppm, adsorbent dosage 0.5 g/25 mL, and mixing speed 200 rpm).

In this work, 98–99% of Cu(II) ions and 95% of Ni(II) ions, respectively, were removed by PKSC and MPKSC. The removal efficiency was higher compared to previous studies, whereby Cu(II) (97% removal), and Ni(II) (55% removal) [26]. However, Cr(IV) removal efficiency by MPKSC (89.2% removal) is lower than previous study using PKS (97% removal) [22]. Removal efficiency of Cr(IV), Cu(II) and Ni(II) by PKSC and MPKSC in comparison with biochar examples from previous studies is depicted in Table 4. Overall, results from this study and previous studies indicates that chemical modification of biochar does not necessarily result in high removal efficiency of Cr(IV) [30], [32].

**Table 4.** Removal efficiency of Cr(IV), Cu(II) and Ni(II) by PKSC and MPKSC in comparison with biochar examples from previous studies.

| Sorbent                    | Cr(VI) Efficiency (%) | Cu(II) Efficiency (%) | Ni(II) Efficiency (%) | Reference |
|---------------------------|-----------------------|-----------------------|-----------------------|-----------|
| Peanut shell biochar      | 89.54                 | -                     | -                     | [29]      |
| Chemically treated peanut shell biochar | 47                    | -                     | -                     | [30]      |
| Citric acid treated peach stone shell | 74.59                 | -                     | -                     | [31]      |
| Chemically treated almond shell | 29.06                 | 36.17                 | -                     | [32]      |
| Biochar-Pistachio Shell   | -                     | 99.9                  | -                     | [33]      |
| Chemically treated ginkgo shell biochar | -                     | 83.6                  | -                     | [34]      |
Walnut shell biochar  -  -  96.0  [35]
Acid treated coconut copra -  -  47.2  [36]
Acid treated Mangosteen shell -  -  99.0  [37]
PKSC  48.98  98.62  97.10  This study
MPKSC  42.97  99.29  96.77  This study

3.4. Adsorption isotherms study
Table 5 shows that Cr(IV) and Ni(II) adsorption by PKSC were best represented by Freundlich isotherm with strong $R^2$ linearity of 0.922 and 0.982, respectively. The Freundlich isotherm indicates that adsorption occurs on heterogeneous surfaces of adsorbent [27]. In this study, calculated $n$ (from $1/n$) for Cr(IV) and Ni(II) were 1.033 and 1.870, respectively, signifying a favourable adsorption process. On the other hand, Cu(II) adsorption by PKSC can explained by using Langmuir isotherm, indicating a monolayer adsorption process. Table 6 shows that the adsorption of Cr(IV), Ni(II) and Cu(II) by MPKSC can be represented by Freundlich isotherm. The plot against log $Q_e$ against log $C_e$ showed excellent $R^2$ values for all studied heavy metals (Cr (IV): 0.924, Ni (II): 0.985, Cu (II):0.931).

Table 5. Langmuir and Freundlich constant for the adsorption of Cr(IV), Ni(II), and Cu(II) by PKSC

| Heavy Metal ions | Langmuir constant | Freundlich constant |
|------------------|-------------------|---------------------|
|                  | $Q_m$  | $b$    | $R^2$   | $K_f$   | $1/n$  | $R^2$ |
| Cr(IV)           | 0.842  | 0.893  | 0.155   | 0.030   | 0.968  | 0.922 |
| Ni(II)           | 3.477  | 0.070  | 0.772   | 0.345   | 0.534  | 0.982 |
| Cu(II)           | 0.200  | -9.30  | 0.724   | 0.219   | -0.844 | 0.136 |

Table 6. Langmuir and Freundlich constant for the adsorption of Cr(IV), Ni(II), and Cu(II) by MPKSC

| Heavy Metal ions | Langmuir constant | Freundlich constant |
|------------------|-------------------|---------------------|
|                  | $Q_m$  | $b$    | $R^2$   | $K_f$   | $1/n$  | $R^2$ |
| Cr(IV)           | -0.701 | -0.019 | 0.700   | 0.007   | 1.408  | 0.924 |
| Ni(II)           | 7.435  | 0.232  | 0.144   | 0.943   | 1.375  | 0.985 |
| Cu(II)           | -0.993 | -0.676 | 0.573   | 1.716   | 1.655  | 0.931 |

3.5. Kinetic study
The pseudo-first-order and pseudo-second-order models were used in kinetic study of heavy metal adsorption by PKSC and MPKSC. The values of the rate constants, $K_1$, and $K_2$, were obtained from the slope of the straight-line graph. A relatively high $R^2$ value (equal or near to 1) specifies that adsorption kinetics are effectively represented by the model [16].

The $R^2$ values shown in Table 7 indicates that PKSC and MPKSC adopted the pseudo-second-order. In comparison, the pseudo-second-order reaction model yielded a very strong straight line from the effects of the fitted data relative to the pseudo-first-order reaction model, which was called a nonlinear model [16]. The pseudo-second-order value of $R^2$ is mostly greater than $R^2 > 0.99$ except Ni (II) from the PKSC study with an $R^2$ correlation coefficient of 0.9086, while the correlation coefficient for the pseudo-first-order model is $R^2$ from 0.6723 to 0.9716.

In a pseudo-second-order reaction, the chemisorption is linked to the diffusional process of the cationic metal ions to the adsorbent surface, and the latter to the ion-exchange mechanism [28]. Heavy metals typically bind to the adsorbent surface through the covalent bonds at all accessible locations, optimizing their coordination number with the surfaces. Overall, the $R^2$ values of the pseudo-second-order kinetic model were higher in contrast to the pseudo-first-order kinetic model. Therefore, the
analysis showed that the adsorption of these heavy metal ions followed the pseudo-second-order kinetic paradigm for the PKSC and MPKSC.

**Table 7.** Pseudo First-order and Pseudo Second-order constants for Cr(IV), Ni(II), and Cu(II) adsorption by PKSC and MPKSC

| Adsorbent | Heavy Metal ions | Pseudo First-order kinetics | Pseudo-second-order kinetics |
|-----------|------------------|-----------------------------|------------------------------|
|           | K₁               | Qₑ₁           | R²               | K₂               | Qₑ₂           | R²               |
| PKSC      | Cr (IV)          | 0.09          | 1.179           | 0.8586           | 1.1118          | 0.300           | 0.9927           |
|           | Ni (II)          | 0.0053        | 1.439           | 0.8474           | 1.0316          | 0.059           | 0.9086           |
|           | Cu (II)          | 0.0004        | 1.506           | 0.9336           | 1.4019          | -0.549          | 0.9983           |
| MPKSC     | Cr (IV)          | 0.0151        | 1.113           | 0.9272           | 0.8757          | -0.100          | 0.9905           |
|           | Ni (II)          | 0.0017        | 1.959           | 0.6723           | 1.1638          | 0.200           | 0.9971           |
|           | Cu (II)          | 0.0004        | 1.519           | 0.9716           | 1.414427        | -2.116          | 0.9991           |

4. Conclusion
Chemical modification of PKS biochar via acid-base treatment has successfully improved its adsorptive properties. The surface area was increased from 112,934 m²/g to 149,670 m²/g by acid-based treatment. Batch adsorption study showed that the MPKSC afforded high removal efficiency for Cu (99.29%), Ni (96.77%) and Cr (42.97%). The Cr(IV) and Ni(II) adsorption by PKSC, as well as Cr(IV), Ni(II) and Cu(II) adsorption by MPKSC were best represented by Freundlich isotherm. However, Cu(II) adsorption by PKSC can explained by using Langmuir isotherm. All studied heavy metals fitted the pseudo-second-order kinetic.

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