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# Removal Efficiency of Cd from Aqueous an Solution by $H_2O_2$ and $KMnO_4$ modified Biochar Derived From Orange and Pea Shells

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## Abstract

Finding an effective and environmentally acceptable adsorbent is crucial for wastewater treating. Biochar is a cheap and environmentally friendly adsorbent for Cd that is produced during the pyrolysis of biomass. Many modification techniques have been employed to increase the adsorption capacity of biochar because it has a low adsorption ability when it is unmodified due to its poorly porous structure and lack of surface functional groups. In this study, two types of biochars (orange-shell biochar and pea-shell biochar) and two modifiers ($H_2O_2$ and $KMnO_4$) were prepared. A batch experiment was carried out to evaluate their removing efficiencies for Cd from a polluted solution. The results showed the adsorption ability of Cd by the above prepared biochar increased with increasing of their used doses and the highest removal efficiency was noticed due to application of 1 and 2 g biochars $L^{-1}$. $KMnO_4$ modified biochars were better in removing of Cd than $H_2O_2$ modified biochars and both modifiers were better in removing Cd than the unmodified ones. The highest eliminating percentages of Cd were 91.75% and 93.10% due to the use of $KMnO_4$ pea-shell biochar at levels of 1 and 2 g biochars $L^{-1}$. In view of our results, the $H_2O_2$ and $KMnO_4$ modification of orange-shell and pea-shell biochars can be considered as a promising and non-cost operative sorbent to remove Cd from water/wastewater

**Keywords:** Biochar, Modified, polluted water, Cd removing

## Introduction

One of the main environmental issues is heavy metal poisoning of water. Heavy metal pollution is the result of heavy metals being released into natural water due to the widespread use and exploitation of fossil fuels, as well as the quick development of metal smelting, battery production, and chemical production (Ahmed et al., 2014; Li et al., 2018). On the other hand, heavy metals can be removed from soil or water, but they are not biodegradable and can travel up the food chain. Lead and cadmium are among the most hazardous heavy metals. The detrimental impacts of cadmium (Cd) on human neurological, skeletal, circulatory, enzymatic, endocrine, immunological, respiratory, and reproductive systems make its widespread presence in the environment concerning (Zhang et al., 2020). These days, a variety of techniques, such as solvent extraction, packed bed filtration, ion-exchange, electrocoagulation, membrane separation, reverse osmosis, electrodialysis, and adsorption, are employed to remove Cd from waste water (Li et al., 2017; Li et al., 2020). Adsorption is one of these strategies that have been shown to be crucial, the most practical, affordable, and widely used for removing Cd from the environment (Peng et al., 2017; Liu et al., 2022). Cd removal from wastewater is currently accomplished using a variety of adsorbent materials, such as silica microspheres and nano-composites (Wang et al., 2019; Dutta and Nath, 2018). Nevertheless, the use and mass-production of these adsorbents remain challenging. Thus, there is an immediate demand for environmentally friendly and reasonably priced adsorbents.

Recently, there has been a surge in interest in bio-sorbents such as biochar, a pyrogenic carbon substance generated from biomass, due to its significant affinity for different types of water pollutants (Yao et al., 2012; Zhou et al., 2014; Wang et al., 2015). Researchers like Mohanty et al. (2018), Rajapaksha et al. (2018), Li et al. (2019) and Pang et al. (2019) have shown that using biochar is thought to be possible for wastewater remediation. It is also thought to be very beneficial for general environmental remediation (Yoo et al., 2018). Comparing it to modified carbon; it is thought to be a superior sorbent material for eliminating heavy metals from water due to its benefits of affordability, accessibility, and affinity (Shakoor et al., 2019). However, because of its low specific surface area and low functional group content on the surface, using biochar directly as an adsorbent to remove different pollutants is not suitable (Li et al., 2017). Consequently, more adjustment is required. Physical and chemical alteration are currently the most widely

used modification techniques (Sajjadi et al. 2019 a,b). Methods for creating tailored biochars that can more effectively sorb heavy metals and other pollutants from water have recently been developed. Either surface modification of pristine biochars or direct pyrolysis of pretreatment biomass feedstocks are used to generate the tailored biochars (Wang et al., 2015; Li et al., 2020). These two techniques, especially the latter, can provide high-efficiency adsorptions for the removal of heavy metals from water since they are not only simple and affordable but also appropriate for large-scale operations (Rajapaksha et al., 2016). The alteration of biochar to increase its usefulness is a new area of research. Techniques for modification include impregnation with minerals, steam activation, base and/or acid treatment, and magnetic modification. It has been observed that chemical modifications made to the biomass or the biochar produced after pyrolysis has a significant impact on the adsorption capacity of biochar (Sarkar et al., 2019). This can be explained by altering the biochar's interfacial region and raising its chemical reactivity through the formation of surface functional groups, which subsequently increases adsorption sites.

Potassium permanganate (KMnO4) has been found to have superior modification capability (Wang et al., 2015). Since $H_2O_2$ breaks down into $H_2O$ and $O_2$, it is a reasonably cheap and clean gas that is unlikely to generate secondary pollution. $H_2O_2$ alteration would therefore be sustainable and inexpensive. While $H_2O_2$ has been utilized to alter hydrochar (Zuo et al., 2016; Wang and Liu, 2018), not much research has been done on how $H_2O_2$ affects the properties and ability of its tailored biochar to adsorb heavy metals.

Our study was conducted on using pea-shell and orange-shell biochars and their modified biochars by $H_2O_2$ and $KMnO_4$ for Cd removal from aqueous media to investigate the removing efficiency of Cd and by these types of biochars. The findings of this work may contribute to the creation of affordable techniques for the removal of Cd, preventing environmental contamination, and improving human health.

## Materials and Methods

### 1. Materials

The experimental vegetable and fruit fields of Benha University's Faculty of Agriculture in Moshtohor village, Toukh City, Qalubiea Governorate, Egypt, provided the pea and orange shells that were gathered. For alternate uses, such feedstocks are readily available, inexpensive, and plentiful in the established markets. The agricultural wastes were dried in an oven at 60-70 °C until they reached a consistent weight after being repeatedly cleaned in deionized water. The dried wastes were ground into 5–10 mm powders by a slow pyrolysis method before being used to produce biochar. For this investigation, analytical-grade chemicals and reagents were used exclusively. The hydrogen peroxide ($H_2O_2$), potassium permanganate (KMnO4), and cadmium nitrate tetrahydrate ($Cd(NO_3)_2.4H_2O$) were purchased from Al Gamohoryea Chemical Reagent Co., Ltd. (Cairo, Egypt).

### 2. Biochar preparation and modification

Biochars from orange-shell and pea-shell were created by gradual pyrolysis in a pyrolysis furnace for two hours at 400 °C. Next, combining 0.5% $KMnO_4$ with biochar was created with a ratio of 2 mL $KMnO_4$: 1 g biochar (Fan et al., 2018). After 4 hours at room temperature, the combination was exposed to air at 60 °C for an additional 4 hours. In order to create $H_2O_2$ modified biochars, a ratio of 20 mL $H_2O_2$, 30%): 3 (3 g of biocahr) were used. Pea-shell and orange-shell biochars were added to $H_2O_2$ (30%) at room temperature (25 °C). After five hours, the biochar was taken out, cleaned with ultrapure water, and dried in an oven at 80 degrees Celsius (Xue et al., 2012). The modified biochars were periodically cleaned with deionized water three times, dried at 60 °C until their weight remained constant, and then sieved through a mesh screen of 100 mesh before being sealed in an airtight bottle for use in batch adsorption tests.

### 3. Biochar analyses

The powdered biomass was tightly placed in a ceramic pot, and then pyrolyzed in a muffle furnace under $N_2$ atmosphere, the pyrolysis was programmed to raise the internal biomass chamber temperature to 400°C at a rate of 5 °C/min and held at the peak temperature for 2 h before cooling to room temperature. All of the biochar samples were ground to pass through a 0.25-mm sieve prior to use (Cui et al., 2016).

The basic physicochemical properties, including the pH, cation exchange capacity (CEC) were determined by the methods described in (Yan et al. (2020). Total elemental composition such as C, O, N, H and S of biochars were measured by elemental analyzer (Vario EL, Elementar). The specific surface areas of biochars were determined using the Brunauer-Emmett-Teller (BET) adsorption method with $N_2$ (Peng et al., 2017). The chemical properties of the used biochars were presented in Table 1.

**Table 1.** Some chemical properties of the used biochars

| Properties | ORB | PSB | ORHB | PSHB | OSKMB | PSKMB |
|---|---|---|---|---|---|---|
| pH | 8.16 | 8.59 | 6.84 | 7.13 | 8.94 | 9.23 |
| CEC (cmole $kg^{-1}$) | 58.7 | 72.8 | 124 | 152 | 187 | 243 |
| SA ($m^2$ $g^{-1}$) | 75.5 | 106 | 185 | 276 | 287 | 356 |
| C % | 63.2 | 71.4 | 52.4 | 60.8 | 47.5 | 51.5 |
| N % | 0.75 | 1.87 | 0.62 | 1.54 | 0.43 | 1.29 |
| H % | 1.85 | 2.01 | 2.32 | 2.67 | 2.91 | 3.43 |
| O % | 11.4 | 15.6 | 20.8 | 26.4 | 31.3 | 36.2 |

OSB= Orange-shell biochar, PSB= Pea-shell biochar, OSHB= Orange-shell $H_2O_2$ biochar, PSHB= Pea-shell $H_2O_2$ biochar, OSKMB= Orange-shell $KMnO_4$ biochar and PSKMB= Pea-shell $KMnO_4$ biochar

**4. Batch adsorption**

Cadmium nitrate (Cd $(NO_3)_2 \cdot 4H_2O$) was s dissolved in distilled water to provide 1000 mg $L^{-1}$ cadmium stock solution. In this experiment, the concentration of Cd (20 mg $L^{-1}$) was set using the dilution method. After that, the batch experiments were performed to assess the adsorption of Cd onto orange-shell and pea-shell biochars and their $H_2O_2$ and $KMnO_4$ modifiers. In order to investigate the impact of biochar mass (varying from 0.1 g to 2 g $L^{-1}$) on Cd adsorption, solutions containing 20 mg Cd per liter were combined with the produced biochars and their modifiers. The mixtures of Cd solution (20 mg $L^{-1}$) and the used biochars and their modifiers were adjusted at a pH of 6 and then were shaken continuously (at 160 rpm) at room temperature (25 ± 0.5 °C) for six hours. Atomic absorption spectroscopy (AAS) was used to determine the amounts of Cd in the filtrates. The removing efficiency (RE) of Cd by the prepared biochars was calculated following this equation: RE (%) = C0-Ce/C0*100, where C0 and Ce (mg/L) represent the concentration of Cd at the initial and equilibrium times, respectively.

**5. Statistical analyses**

All experiments were conducted with four replicates and the collected data were statistically analyzed with one way analysis of variance using SPSS 20 software. The multiple-range test of Duncan was used to compare the mean values of the data from each of the treatments at $p < 0.05$.

**Results**

Data in Figures 1-3 showed that the efficiency of unmodified biochars (orange-shell biochar and pea-shell biochar) and their modifiers ($H_2O_2$ orange-shell biochar, $H_2O_2$ pea-shell biochar, $KMnO_4$ orange-shell biochar and $KMnO_4$ pea-shell biochar) on Cd removal from an artificial polluted solution. The effect of modified biochars in removing Cd from the solution was higher than that of unmodified biochars and the removing ability of these materials significantly increased with increasing of their applied masses. For the mean effect of biochar types, it was found that $KMnO_4$ pea-shell modified biochar led the highest decreases in Cd concentrations with a percentage of 83.77% followed by $KMnO_4$ pea-shell modified biochar with a percentage of 75.2% with a significant difference between them (Fig.1).

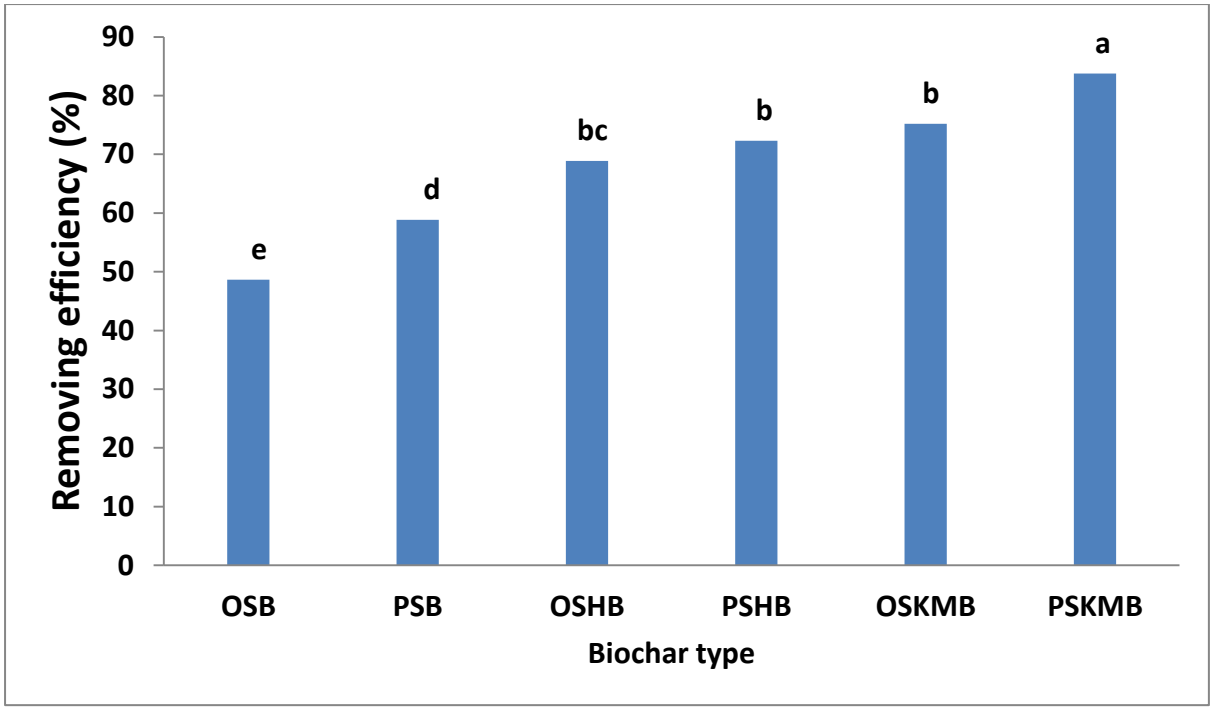


Fig. 1. Mean effect of biochar type on removing of Cd.
OSB= Orange-shell biochar, PSB= Pea-shell biochar, OSHB= Orange-shell $H_2O_2$ biochar, PSHB= Pea-shell $H_2O_2$ biochar, OSKMB= Orange-shell $KMnO_4$ biochar and PSKMB= Pea-shell $KMnO_4$ biochar

For evaluating the mean effect of biochars masses, the results indicated that using 1g and 2 g per liter of the prepared biochars and their modifiers were responsible for the largest removing efficiency for Cd from the polluted solution with percentages of 77.21% and 78.89%, respectively without a noteworthy change between them (Fig. 2).

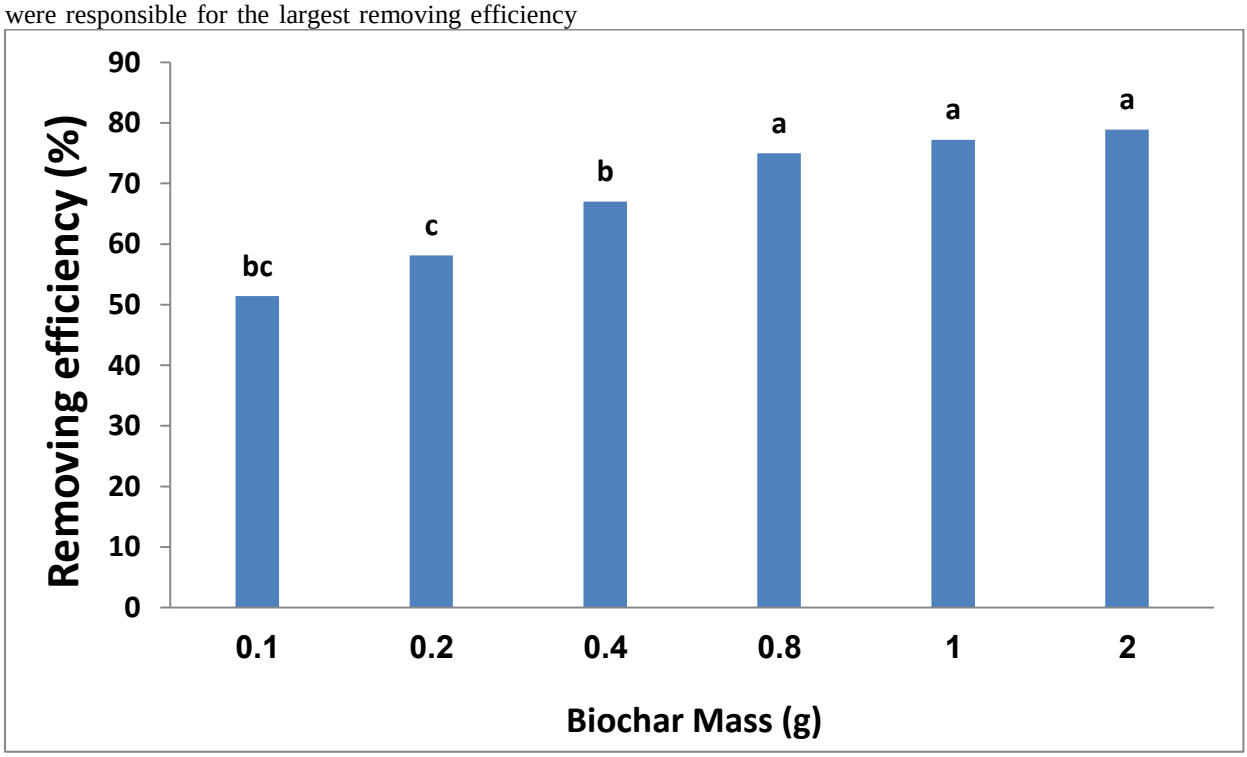


Fig. 2. Mean effect of biochar mass on removing of Cd.

OSB= Orange-shell biochar, PSB= Pea-shell biochar, OSHB= Orange-shell $H_2O_2$ biochar, PSHB= Pea-shell $H_2O_2$ biochar, OSKMB= Orange-shell $KMnO_4$ biochar and PSKMB= Pea-shell $KMnO_4$ biochar

In the case of evaluating the interaction between biochar types and different applied masses of biochars, our finding in Fig 3 showed that the removing efficiency were ranged 32.75% to 57.25%

for orange shell biochar, from 44.70% to 68.95% for pea shell biochar, from 50.65% to 82.45% for $H_2O_2$ orange shell biochar, from 54.40% to 82.25% for $H_2O_2$ pea shell biochar, from 59.20 to 86.85% for $KMnO_4$ orange shell biochar and from 66.95% to 93.10% for $KMnO_4$-pea shell biochar. These results indicated the removing efficiency of Cd was taken the following order: $KMnO_4$ pea-shell biochar> $KMnO_4$ orange-shell biochar> $H_2O_2$ pea-shell biochar> $H_2O_2$ orange-shell biochar> pea-shell biochar> orange-shell biochar.

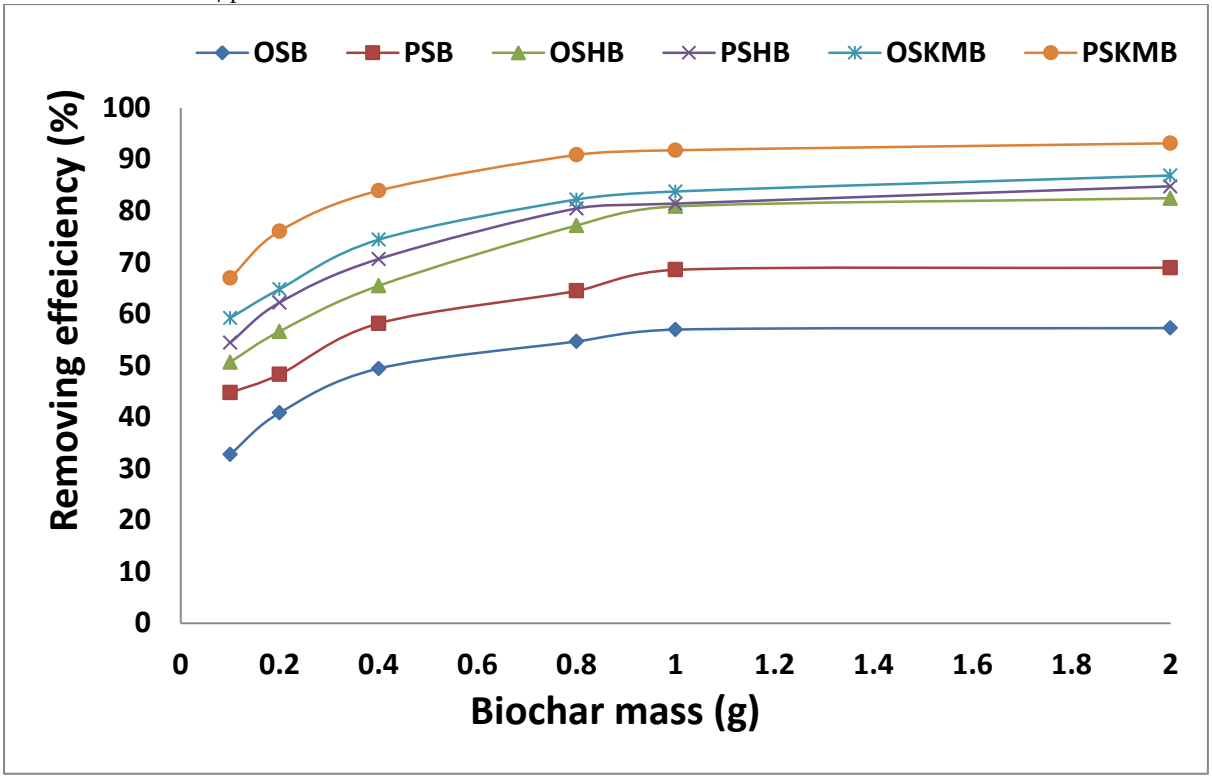


Fig. 3. Interaction effect of biochar mass and biochar type on removing of Cd.
OSB= Orange-shell biochar, PSB= Pea-shell biochar, OSHB= Orange-shell $H_2O_2$ biochar, PSHB= Pea-shell $H_2O_2$ biochar, OSKMB= Orange-shell $KMnO_4$ biochar and PSKMB= Pea-shell $KMnO_4$ biochar

## Discussion

The biochars surface areas were doubled following the modification process. In our study, orange shell biochar and pea shell biochar had surface areas of 68 and 115 $m^2/g$ but the modified biochars had surface areas of 135, 176, 198 and 245 205 $m^2/g$ for $H_2O_2$-orange shell biochar, $H_2O_2$-pea shell biochar, $KMnO_4$-orange shell biochar and $KMnO_4$-pea shell biochar, respectively. Our results are in agreement with the findings of Wang et al. (2015) who indicated that adding potassium permanganate to the biochar enhanced its surface qualities, making it more suitable for use as an adsorbent in environmental applications. Moreover, the removal rate continued to increase slightly with each subsequent dosage increment. The clearance rates of Cd rose from 63.1% to 93.2%, when the dose of designed biochar was raised from 0.1 to 2 g/L. One of the following scenarios could explain how Cd sorption occurs on biochar: (i) Cd precipitate with minerals (such $PO_4^{3-}$ and $CO_3^{2-}$). (Zhang et al., 2013, 2015); (ii) metal ion exchange with $Cd^{2+}$ ($K^+$, $Ca^{2+}$, $Na^+$, $Mg^{2+}$, –COOM, –R–O–M) (Ahmad et al., 2014;); (iii) surface complexation between Cd2+ and oxygen-containing functional groups (e.g., –OH, –R–OH, –COOH) (Sun et al., 2016; Wan et al., 2014); and (iv) coordination of $Cd^{2+}$ with π electrons (e.g., C=C) (Zhang et al., 2015). The suggested mechanisms could be attributed to the pH, ionic strength, and surface features of biochar that are temperature-dependent during pyrolysis (Uchimiya, 2014).

According to the after mentioned assessments, chemically modifying biochar has emerged as a significant technique to increase its environmental uses, particularly in the removal of heavy metals from water. Li et al. (2017) investigated the adsorption of Cd from aqueous solutions by KMnO4 impregnated biochar (BC-MnOx). Their findings demonstrated that, following impregnation, manganese oxide particles were successfully distributed throughout the biochar; the addition of KMnO4 enhanced the micropore composition, boosted surface area, and created inner-sphere composites with the biochar's oxygenic functional groups. The elimination (%) of Cd rose progressively with dosage enhancement, but the adsorption capacity of Cd showed a reverse trend (Zhang et al., 2022). The reason given was that saturated biochar was unable to provide more adsorption sites for

subsequent Cd adsorption at a reduced dosage. According to Wang and Liu (2018), the removal efficiency of metal ions like Cd increased significantly following $H_2O_2$ modification because the carboxyl functional groups increased. They also suggested that $H_2O_2$ modification led to decreased precipitation, which allowed the increased oxygen-containing functional groups to react with more metal ions. Ultimately, complexation was responsible for the majority of the Cd removal. In summary, the kinds of oxidant and biochar used have a significant impact on the removal capability of Cd.

## Conclusion

Orange shell and pea shell biochars modified by $H_2O_2$ and KMnO4- are markedly successful in removing Cd professionally. This confirms that the chemical pretreatment method can be used to prepare high competence biochars for Cd removing from polluted solutions. Results also concludes that KMnO4 modified orange shell and pea shell biochars were more efficient in removing Cd from the polluted solution than $H_2O_2$ modified orange shell and pea shell biochars and also than the unmodified. To expand the usefulness of these resultant sorbents in wastewater treatment and to develop novel low-cost and high-efficiency modification processes for biochar, more research is still required. Therefore, additional in situ research is required to clarify the true impact of biochar on the environment.

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# كفاءة إزالة الكادميوم من محلول المائي بواسطة الفحم الحيوي المشتق من قشور البرتقال والبازلاء والمعدل بواسطة $H_2O_2$ و$KMnO_4$

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قسم الأراضي والمياه – كلية الزراعة بمشتهر – جامعة بنها

يعد العثور على مادة ماصة فعالة ومقبولة بيئيًا أمرًا بالغ الأهمية لمعالجة مياه الصرف الصحي. Biochar عبارة عن مادة ماصة رخيصة الثمن وصديقة للبيئة والتي يتم إنتاجها أثناء الانحلال الحراري للكتلة الحيوية. تم استخدام العديد من تقنيات التعديل لزيادة قدرة امتزازالفحم الحيوي لأنه يتمتع بقدرة امتصاص منخفضة عندما لا يتم تعديله بسبب بنيته المسامية الضعيفة ونقص المجموعات الوظيفية السطحية. في هذه الدراسة تم تحضير نوعين من الفحم الحيوي (فحم من قشور البرتقال وفحم حيوي من قشور البازلاء) واثنين من المعدلات ($H_2O_2$ و$KMnO_4$). تم إجراء تجربة دفعة لتقييم كفاءتها في إزالة الكادميوم من المحلول الملوث. أظهرت النتائج أن قابلية امتزاز الكادميوم بواسطة الفحم الحيوي المحضر أعلاه تزداد مع زيادة الجرعات المستخدمة، كما لوحظت أعلى كفاءة إزالة عند إضافة 1 و 2 جرام من الفحم الحيوي $L^{-1}$. كان الفحم الحيوي المعدل بـ $KMnO_4$ أفضل في إزالة الكادميوم من الفحم الحيوي المعدل بـ $H_2O_2$ وكان كلا المعدلين أفضل في إزالة الكادميوم من الفحم الحيوي غير المعدل. وكانت أعلى نسب إزالة للكادميوم هي 91.75% و93.10% بسبب استخدام الفحم الحيوي لقشرة البازلاء $KMnO_4$ عند مستويات 1 و2 جرام من الفحم الحيوي $L^{-1}$. في ضوء النتائج التي توصلنا إليها، يمكن اعتبار تعديل $H_2O_2$ و$KMnO_4$ للفحم الحيوي لقشور البرتقال و البازلاء مادة ماصة فعالة واعدة وغيرمكلفة لإزالة الكادميوم من المحاليل الملوثة.