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# Efficient Removal of Multiple Heavy Metals from Simulated Wastewater using Combined Carbonized Palm Shell and Rice Husk Adsorbents in Multi-Column System

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**Abstract:**
This research study amid to develop a hybrid-modified adsorbent from carbonized palm shell and rice husk to remove heavy metal components from simulated wastewater using multi-bed column adsorption. Physical treatment for the adsorbents was done before conducting the experiments. Removal of 13 heavy metal components were investigated in this work and the adsorption performance was compared to that of single bed column adsorption. Six types of adsorbents, including one pure blended rice husk adsorbent, one pure carbonized palm kernel shell adsorbent, and four hybrid adsorbents with different mixing ratios (20%, 40%, 60%, and 80%wt) of blended rice husk and carbonized palm kernel shell were used in this study. Surface morphology analysis was conducted using Field Emission Scanning Electron Microscope (FESEM) for each adsorbent, and adsorption performance was evaluated by area under the graph analysis. The results showed that cadmium, copper, vanadium, antimony, and zinc were effectively adsorbed by the hybrid adsorbent compared to single adsorbents. The adsorption capacity was increased in multi-layer bed adsorption, and the hybrid adsorbent with multi-bed layer column performed better than single-layer bed column. In conclusion, a hybrid adsorbent of mixing of rice husk and carbonized palm shell and applying multi-bed layer column adsorption contributed to enhanced adsorption performance of removing multiple heavy metals from wastewater.

**Keywords**: Adsorption, Multi-heavy metals, wastewater, Hybrid adsorbents.

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# إزالة فعالة للمعادن الثقيلة المتعددة من المياه العادمة باستخدام نظام متعدد الأعمدة مع مواد ماصة مخلوطة من قشور النخيل والأرز المعدلة والمفحمة

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## الملخص

الغرض من هذه الدراسة هو تحضير مادة ماصة معدلة هجينة من قشرة نواة النخيل المتفحمة وقشر الأرز عن طريق المعالجة الفيزيائية لإزالة سلاتة عشر عنصر تقيل من المياه العادمة بواسطة عمود الامتزاز المتعدد الحشوات ومقارنة معدل أداء الامتصاص باستخدام عمود الامتزاز الأحادي الحشوات. تم استخدام ستة أنواع من المواد المازة لإزالة المعادن وهي مادة مازة محضرة من قشر الأرز المخلوط الصافي (100%)، وأخرى من قشور نواة النخيل المتفحم (100 %)، وأربعة مواد مازة هجينة محضرة بخلط قشر الأرز المخلوط وقشر نواة النخيل المتفحمة بنسب خلط 20 %، 40%، 60%، 80% نسبة وزنية بالنسبة إلى قشر الأرز المخلوط. تم تقييم خصائص كل مادة ممتزة بشكل فردي بتحليل المورفولجي للسطح باستخدام مجهر المسح الإلكتروني للانبعاثات. تم تقييم أداء الامتزاز لكل مادة ممتزة عن طريق تحليل المساحة تحت منحنى الرسم البياني. أشارت النتائج التي تم الحصول عليها بالنسبة لتحليل المساحة الفردية تحت المنحنى إلى أن عناصر كل من الكادميوم والنحاس والفاناديوم والرصاص والأنتيمون والزنك أكثر جاذبية للامتزاز باستخدام خلط قشر الأرز وقشرة النخيل الكربونية الممزوجة الهجينة كمادة مازة في عمود الامتزاز المتعدد الحشوات مع زيادة قدرة الامتزاز مقارنةً بنفس الممتزات المستخدمة بالامتزاز باستخدام عمود الامتزاز الأحادي الحشوات. لذلك، يمكن استنتاج أن خلط قشر الأرز وقشرة النخيل المتفحمة كممتز هجين شارك في تعزيز أداء امتزاز عناصر الكادميوم والنحاس والفاناديوم والرصاص والأنتيمون والزنك، وكذلك تطبيق عمود الامتزاز المتعدد الحشوات لامتزاز عديد المعادن الثقيلة زاد من معدل أداء الامتزاز بالمقارنة مع تطبيق عمود الامتزاز الأحادي الحشوات.

**الكلمات المفتاحية:** الامتصاص، المعادن الثقيلة، المياه العادمة، قشور النخيل المفحمة.

## Introduction

The rise of industry has caused significant harm to the environment, most notably through the depletion and disruption of natural resources and loss of biodiversity. One of the significant environmental problems caused by industrial activities is water pollution resulting from the release of heavy metals such as iron, zinc, copper, chromium, mercury, and lead into water bodies. Many life forms suffer from the toxic effects of these heavy metals, which tend to bioaccumulate in their tissues, and do not degrade into harmless end products. Therefore, their removal from wastewater is necessary to comply with environmental regulations and for the preservation of the environment, human health, and safety [1].

Several treatment methods such as electrolytic recovery, chemical precipitation, ion exchange, and membrane separation have been employed to remove heavy metals from wastewater [1]. However, these methods have several constraints, including high-cost expenditure, inadequate efficiencies at low metal concentrations, and generation of toxic sludge [2]. Therefore, alternative methods that are efficient, cost-effective, and environmentally friendly are required. Adsorption is a promising and highly effective treatment method due to its simple design and sludge-free environment.

Among adsorbents for heavy metal removal, activated carbon reigns supreme due to its vast surface area and intricate network of microscopic pores, facilitating efficient adsorption. However, it suffers from major drawbacks, including being impractical in small and medium industries, expensive materials, and the loss of 10-15% during regeneration process [3]. Therefore, alternative low-cost and natural materials are needed to adsorb water pollutants in wastewater streams. Natural adsorbents derived from agricultural wastes, such as rice husk and oil palm sell, have strong capabilities to adsorb water pollutants [4,5].

However, the effectiveness of raw agriculture waste as adsorbents can be hampered by limitation like low adsorption capacity [6,7]. Therefore, pre-treatment or modification of these materials is essential to maximize their effectiveness in heavy metal decontamination applications. Previous researchers have focused on removing one type of element using agricultural waste materials [8-12], while very few studies have been conducted on the removal of multi-heavy metals from wastewater [13,14]. In reality, there are many types of elements in wastewater, and there is a lack of research on the removal of multi-metal from wastewater.

Several studies, including those by Kumar and Bandyopadhyay, Hosseinnia et at., and Ong et al., [15-17] have demonstrably advanced the understanding of adsorption in fixed-bed columns using rice husk. In particular, there is a need to investigate the effect of the number of beds via multi-bed column adsorption for the removal of multi-metal components from simulated wastewater.

This study investigated the development of a novel hybrid adsorbent, combining physically treated carbonized palm shell and rice husk, for the removal of multiple heavy metals from wastewater using multi-bed column system. The adsorption performance will be evaluated and compared to prior research employing single-layer fixed-bed columns. While there have been previous studies on the use of adsorbents for heavy metal removal, the novelty of this work lies in the development of a hybrid modified adsorbent from two different sources, which has not been extensively studied in previous literature. The study showed that the adsorption performance of each element is not consistent and can be affected by competition factors in multi-component adsorption. Additionally, the study demonstrate that multi- layer bed adsorption can enhance the adsorption performance of hybrid adsorbents by overcoming the limitation of single-layered fixed bed columns.

## Material and methods

### 1. Materials

This study utilizes wastewater as the target contaminant and explores the application of rice husk and palm kernel shell- based adsorbents for its treatment.

### 2. Methods

### 2.1 Preparation of simulated wastewater

To create a simulated wastewater, a mixture of various chemicals was prepared, including iron (II) sulfate and cadmium chloride, copper (II) nitrate trihydrate, lead nitrate, chromium (VI) oxide, magnesium chloride, manganese (IV) oxide, zinc sulfate, and aluminum chloride. To achieve uniform mixing of the wastewater components, 1g of each chemical was precisely weighed and added to 250 ml of distilled water. The solution was then subjected to vigorous magnetic stirring at 2000 rpm for 5 minutes. The resulting solutions were then combined in a plastic container and diluted with distilled water until the total volume reached 10 L. The mixture was stirred using an electric mixer at 2000 rpm for two hours and then stored in a sealed chemical container.

### 2.2 Preparation of Hybrid Adsorbent combining Physically Treated Palm Kernel Shell and Rice Husk

Hybrid adsorbents were created using rice husk and palm kernel shell adsorbent. To prepare the raw rice husk, it was washed with distilled water and filtered using a GAST Diaphragm Vacuuum pump. It was then dried in a MEMMERT Universal Oven at a temperature range of 105 – 110 °C for 24 hours. Next, the rice husk underwent a physical treatment where it was blended using a Waring Commercial Laboratory Blender to reduce the size. It was then sieved to a size of 150 - 250 µm using a Retsch Mechanical sieve Shaker. The blended and sieved rice husk was washed with distilled water by stirring at 600 minutes and then filtered prior to drying. The resulting ground rice husk stored in a closed plastic bottle.

For the raw palm kernel shell, it was washed and filtered to remove dirt and impurities. After that it was dried in a Memmert Universal Oven at a temperature range of 105 – 110 °C for 24 hours. The palm kernel shells were then carbonized at 600°C in a furnace for 5 hours, followed by size reduction through grinding using a Waring Commercial Laboratory blender and sieving to a desired mesh size range of 150 to 250 µm. The resulting ground carbonized palm kernel shell was stored in a closed bottle.

A total of six adsorbents were prepared and used in single bed column adsorption experiments. Two of the adsorbents were pure blended rice husk (100% BRH) and pure carbonized palm kernel shell (100% CPS), while the other four adsorbents were prepared by mixing blended rice husk and carbonized palm kernel shell with mixing ratios of 20%, 40%, 60%, and 80%wt blended rice husk. The amounts of BRH and CPS used in preparing the adsorbents are shown in table 1 below

**Table 1** Amounts of blended rice husk and carbonized palm shell used in preparing the hybrid adsorbents.

| Adsorbent Composition | Amount of Blended Rice Husk($g_m$) | Amount of carbonized palm shell($g_m$) |
|---|---|---|
| 100%RH | 6 | 0 |
| 80%RH | 4.8 | 1.2 |
| 60%RH | 3.6 | 2.4 |
| 40%RH | 2.4 | 3.6 |
| 20%RH | 1.2 | 4.8 |
| 100% CPS | 0 | 6 |

### 2.3 Screening Analysis of Simulated Water

The Thermo Scientific Inductively Coupled Plasma (ICP) Spectrometer was utilized to measure the concentration of multi-metal elements in simulated wastewater sample. To do this, an 8 ml of the simulated wastewater was extracted from 10 liter plastic chemical container and analyzed.

### 2.4 Characterization of Carbonized Palm Shell and Rice Husk Adsorbents

The adsorption capacity of the adsorbents used in this study is a complex function of various factors, including porosity, total surface area, internal pore volume, and carbon content. To gain a deeper understanding of the performance of the adsorbents, multiple characterization techniques are often employed. In this study, the adsorbents were subjected to a surface morphology analysis, which provided important insights into their microstructure. Field Emission Scanning Electron Microscopy (FESEM) was utilized to study the surface morphology of the rice husk and palm shell adsorbents. The FESEM studies were conducted using a state-of-the-art Zeiss SUPTA 40VP FESEM, which operated at an electron acceleration voltage of 1 kV and a magnification of 1000 times. By examining the surface morphology of the adsorbents, we were able to better understand their properties and potential for effective metal ion adsorption.

### 2.5 Fixed Bed Adsorption Study

The purpose of this series of experiments was to evaluate the performance of multi-layered fixed bed columns for adsorbing multi-metal elements from simulated wastewater. The fixed bed adsorption column was constructed at the laboratory scale, using a 250 ml burette with a diameter of 1.6 cm as the column for backing. To ensure a proper arrangement, a cotton ball with thickness of 1 cm was placed inside the column at the bottom, extending up to 20 cm above the stopcock. Each layer was packed with 2 grams of adsorbent, and the column was packed with three layers of adsorbent, with a fixed distance of 4 cm between each layer.

The column was charged with the simulated wastewater solution in down flow mode manually, with constant fixed flow rate and no pH adjustment was made. To prevent the spread of adsorbent during the filling process, the top surface of the column was covered with a 1 cm thick cotton ball. Treated wastewater samples were collected periodically until each sample reached a volume of 8 ml. The concentration of heavy metals in these samples was then measured using Inductively Coupled Plasme Optical Emission Spectrometry (ICP-OES).

The experiments were conducted at room temperature. The setups were considered semi-batch due to the continuous flow of wastewater entering and exiting the system, while the adsorbent itself remained stationary within the column. The pressure inside the column was maintained at atmospheric pressure. Key parameters such as the inflow rate of wastewater, adsorbent mass, and collected wastewater volume at specific intervals were kept constant throughout the studies. A detailed illustration of the multi-layer fixed-bed column setup is provided in Figure 1.

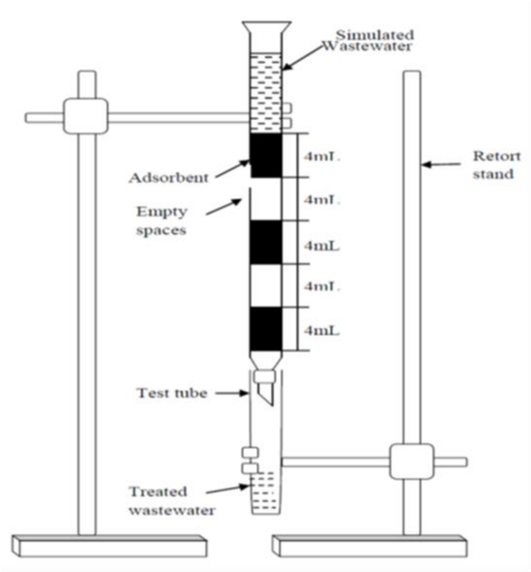


**Figure 1:** Experimental setup for multiple bed adsorptions.

### 2.6 Area under the Graph Analysis

After adsorption, the residual metals concentrations in the treated wastewater samples were analyzed using Thermo Scientific ICP Spectrometer. The performance of the adsorption experiments was evaluated by analyzing the area under the graph using the trapezoidal rule. Since the number of intervals during the 180-minute study was different for each adsorbent, the non-uniform trapezoidal rule was used. Equation 1 was used to calculate the area under the curve.

$$\int_a^b f(x)dx \approx \frac{1}{2}\sum_{k=1}^{N}\left(x_{k+1}-x_k\right)\left(f\left(x_{k+1}\right)+f\left(x_k\right)\right) \quad 1$$

Where a and b represented the values at first and last time intervals, respectively, N is the total number of intervals, and k represents the interval number.

Graphs of element i ($C_i$) as a function of contact time (t) were plotted for each adsorption performance, such that a smaller area, measured under the graph, indicated higher adsorption capacity, while a large area indicated lower adsorption capacity.

## Results and discussion

### 1. Screening Analysis of Simulated Water

Before conducting the adsorption experiments, the initial concentrations of multi-metal elements in the simulated wastewater sample were determined using ICP spectrometer. The following concentrations were obtained: Cadmium (cd) at 68.3002 ppm, Chromium (Cr) at 45.109 ppm, Copper (Cu) at 80.9177 ppm, Iron (Fe) at 8.04997 ppm, Magnesium (Mg) at 33.0103 ppm, Manganese (Mn) at 0.786 ppm, Lead (Pb) at 0.0304424 ppm, Lithium (Li) at 0.98061 ppm, Molybdenium (Mo) at 2.75201 ppm, Nickel (Ni) at 0.0159597 ppm, Antimony (Sb) at 0.114958 ppm, Vanadium (V) at 2.51915 ppm, and Zinc (Zn) at 29.6202 ppm. These concentrations were used as reference for all subsequent adsorption studies. Additionally, the screening analysis of the simulated wastewater revealed a high toxicity of metal elements.

### 2. Surface Morphology Analysis of Rice Husk and Palm Shell Adsorbents

The micrographs presented in figures 2 to 5 reveal significant alterations in the morphology of the rice husk and palm shell adsorbents as a result of the physical treatment they underwent.

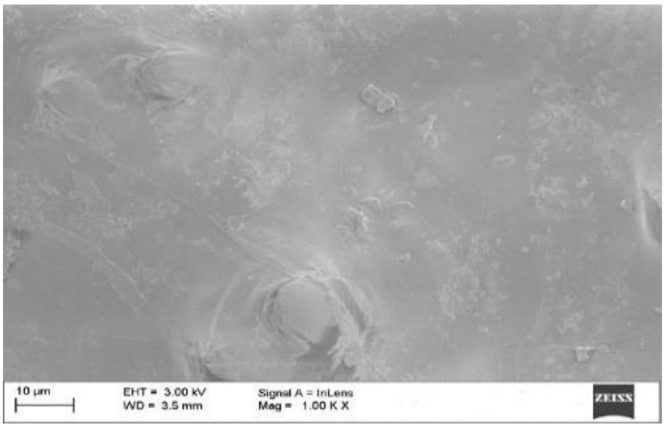


**Figure 2:** Micrograph of the Raw Rice Husk

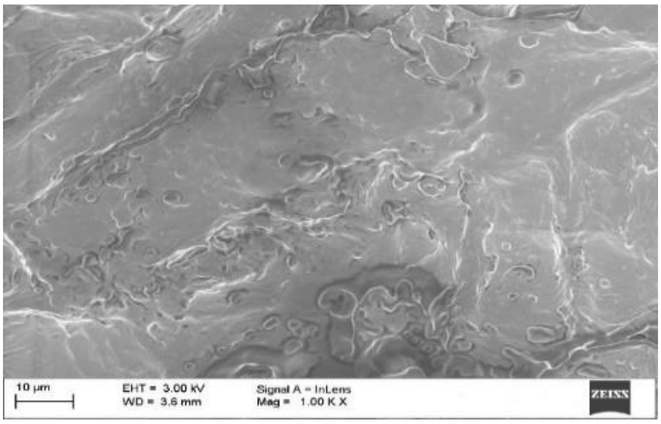


**Figure 3:** Micrograph of the Raw Palm shell

The micrographs in figures 2, 3 show that the surface of raw husk and palm shell adsorbents are covered with a thin layer, which may affect the adsorption performance due to limited space for the adsorption of multi-metal elements. This can result in lower adsorption capacity and efficiency. However, after carbonization, the pore become visible, particularly in the micrograph of carbonized palm shell shown in figure 4. The carbonization process also eliminates volatile compounds and moisture, generating a clear surface with a larger surface area for adsorption of multi-metal elements. This indicates that carbonization enhances the adsorption performance of palm shell adsorbent.

Moreover, the blended rice husk shown in figure 5 exhibited a rougher and more heterogeneous surface compared to the raw rice husk in figure 2. This suggests that the size reduction process in preparing the blended rice husk adsorbent led to an increase in the exposed surface area, providing more sites for the adsorption of multi-metal elements. Therefore, the blended rice husk may have higher adsorption capacity and efficiency than the raw rice husk.

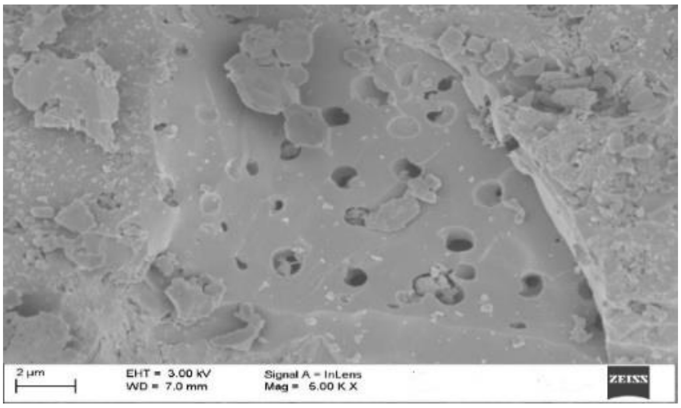


**Figure 4:** Micrograph of the Carbonized Palm shell**.**

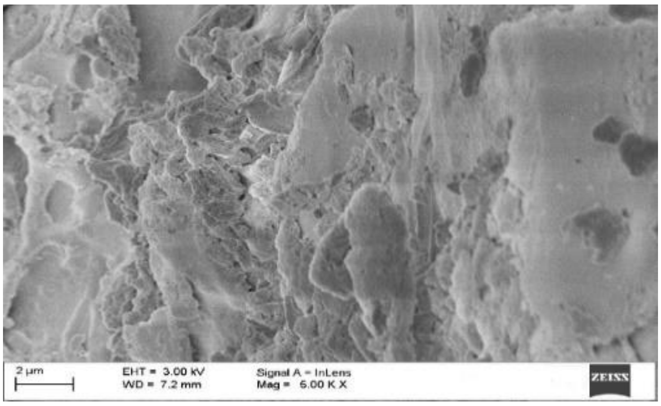


**Figure 5:** Micrograph of the Blended Rice Husk.

In summary, the micrographs show that physical and chemical treatments of the adsorbents can significantly affect the morphology and surface characteristics of the adsorbents, which can ultimately influence their adsorption performance. Carbonization and size reduction were found to enhance the adsorption performance of palm shell and blended rice husk adsorbents, respectively, by increasing the surface area available for adsorption. These findings highlight the importance of optimizing the preparation process of adsorbents to improve their adsorption performance.

## 3. Area under the Graph Analysis

The area under the curve analysis performed in this study provided valuable insights into the adsorption performance of the different types of rice husk and palm shell hybrid adsorbents. Figures 6 (a - m) show the graphs of concentration of metal element *i* ($C_i$) in the treated wastewater at contact time (*t*). It was observed, as apparent in Table 2, that the individual area under the curve values for each metal element were varied and did not follow a consistent pattern due to competition factors in multi-component adsorption. This finding is in agreement with previous studies conducted by Assadek [18].

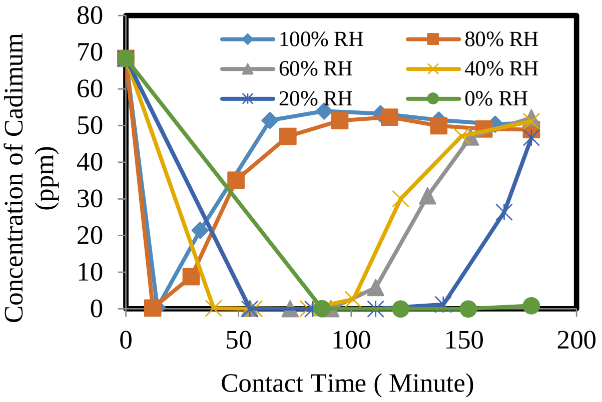
The results summarized in Table 2 showed that some elements such as cadmium, copper, vanadium, lead, antimony, and zinc were more effectively adsorbed by the mixing of rice husk and carbonized palm shell adsorbent compared to pure blended rice husk and pure carbonized palm shell adsorbents in multi-layer bed adsorption. This may be due to the high interaction generated between the metal ion and the hybrid adsorbent surface. Conversely, elements such as chromium, iron, and nickel were better removed with high adsorption capacity by applying pure carbonized palm shell and pure blended rice husk adsorbents. This may be due to the metal ion being more attractive towards interaction with the pure surface adsorbent, such as iron iron towards pure rice husk adsorbent surface and chromium towards pure carbonized palm shell adsorbent surface. Interestingly, certain elements like lithium, magnesium, and molybdenum exhibited minimal removal by most adsorbents, which only occasional low-capacity uptake observed for pure carbonized palm shell. This limited removal efficiency likely stems from a weak interaction between these metal ions and the adsorbent surface. All these findings were similar to our previous study conducted by Assadek 18].

**Table 1** Area under the Graphs for Multi-layer Bed Adsorption Studies by Different Types of Rice Husk and Palm Shell Hybrid Adsorbents.

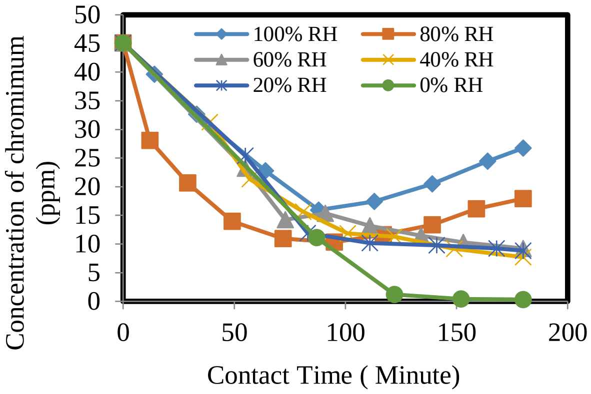
| **Element** | **Adsorbent** | | | | | |
|---|---|---|---|---|---|---|
| | **Area under the curve** | | | | | |
| | **0% RH** | **20%RH** | **40%RH** | **60%RH** | **80%RH** | **100%RH** |
| Cadmium | 2985.473 | 2708.367 | 4273.243 | 4438.853 | 7283.442 | 7861.02 |
| Chromium | 2695.972 | 3434.778 | 3461.179 | 3520.934 | 2899.513 | 4484.61 |
| Copper | 3536.896 | 2232.718 | 1590.653 | 2261.717 | 6160.705 | 6665.49 |
| Iron | 354.3297 | 244.0485 | 217.846 | 319.973 | 70.8685 | 57.46 |
| Lithium | 182.5863 | 147.412 | 146.2853 | 142.8281 | 143.0785 | 129.92 |
| Magnesium | 6213.706 | 9079.435 | 11740.36 | 11719.34 | 9925.49 | 8255.85 |
| Manganese | 56.05523 | 597.6084 | 782.7432 | 793.5907 | 585.5954 | 956.43 |
| Molybdenum | 119.7124 | 582.1029 | 975.1273 | 1317.987 | 1125.845 | 1361.58 |
| Nickel | 2.50056 | 2.25674 | 3.76949 | 4.628055 | 4.377475 | 5.610 |
| Lead | 4.199555 | 1.0145 | 0.69255 | 1.01485 | 0.1824 | 2.64 |
| Antimony | 5.010685 | 4.193997 | 5.625231 | 6.330495 | 7.707258 | 8.64 |
| Vanadium | 156.3709 | 155.8649 | 166.9397 | 187.591 | 161.7049 | 178.92 |
| Zinc | 1299.138 | 996.7863 | 2178.688 | 2452.358 | 4078.871 | 4833.88 |
| | **Total = 17,611.95** | **Total = 20,186.59** | **Total = 25,543.15** | **Total = 27,167.14** | **Total = 32,447.38** | **Total = 34,802.088** |

Furthermore, this study demonstrated a clear advantage for multi-layer bed columns compared to single-layer setups in terms of adsorption performance. This superiority stems from the ability of multi-layer designs to address a critical limitation of single layers: the presence of voids (empty spaces) within the bed. These voids can lead to uneven distribution of wastewater flow throughout the single layer. This uneven flow reduces the chance of contaminant molecules encountering available sites for adsorption. By dividing the column into multiple layers, the wastewater droplets are effectively redistributed as they enter each new layer. This optimized distribution minimizes channeling (preferential flow paths) and pressure buildup within the column, ultimately leading to a significant increase in overall adsorption efficiency.

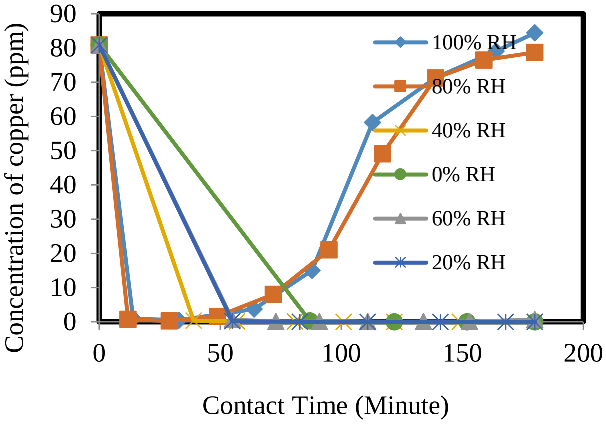
It is important to note that the results of single bed layer adsorption should be selected and optimized in order to find the optimum mixing ratio that minimize the total area under the curve. This optimum ratio can then be used for multi-layer adsorption to produce the highest adsorption capacity of total element adsorption. Therefore, future studies should consider this optimization technique to achieve the best possible adsorption performance.



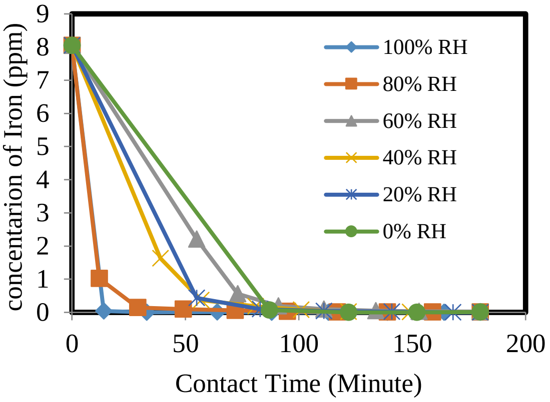
**(a)**



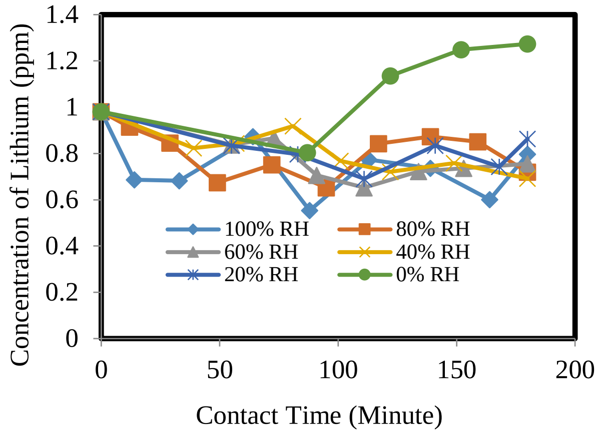
**(b)**



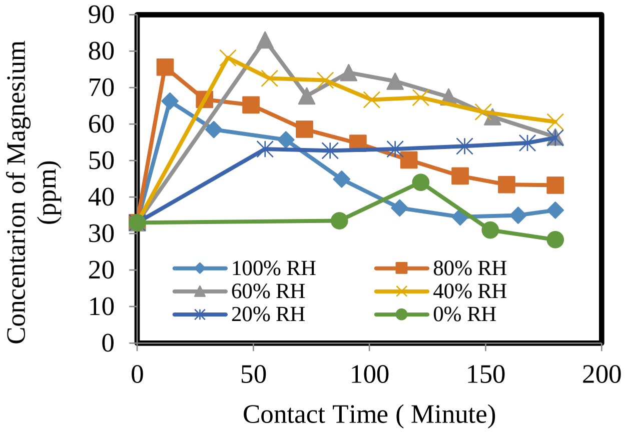
**(c)**



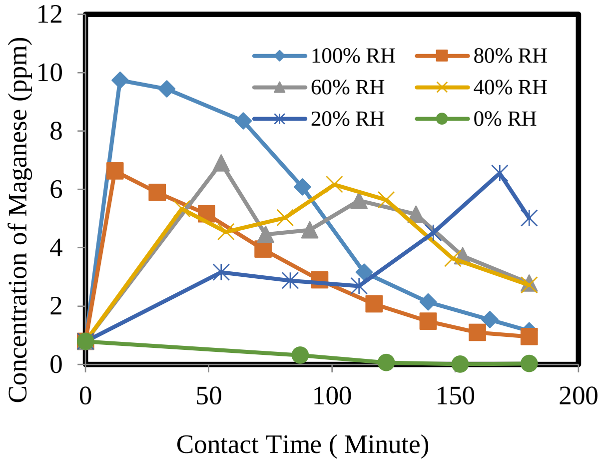
**(d)**


Concentration of Lithium (ppm)
Contact Time (Minute)
0
0.2
0.4
0.6
0.8
1
1.2
1.4
0
50
100
150
200
100% RH
80% RH
60% RH
40% RH
20% RH
0% RH

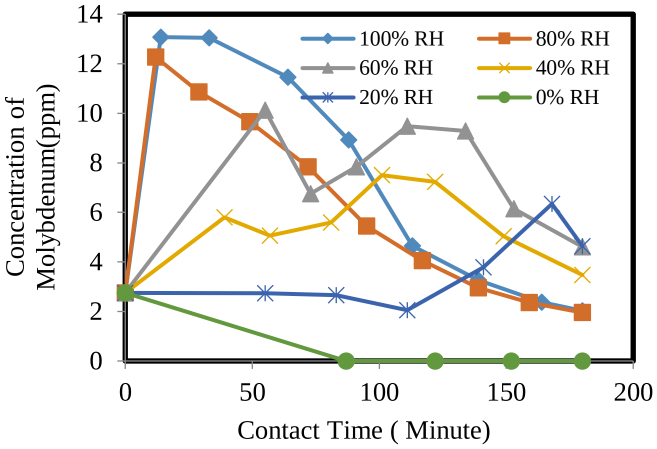
(e)


Concentarion of Magnesium (ppm)
Contact Time ( Minute)
0
10
20
30
40
50
60
70
80
90
0
50
100
150
200
100% RH
80% RH
60% RH
40% RH
20% RH
0% RH

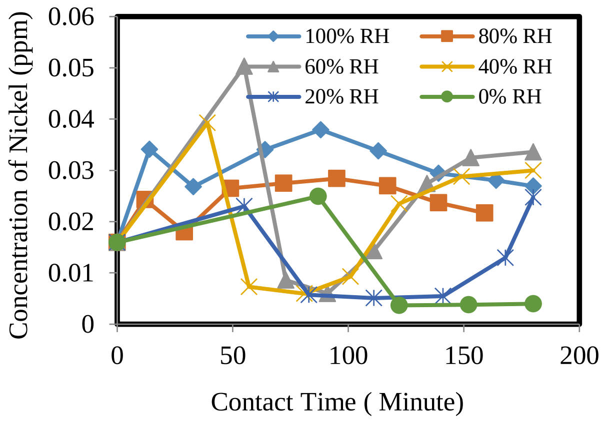
(f)


Concentration of Maganese (ppm)
Contact Time ( Minute)
0
2
4
6
8
10
12
0
50
100
150
200
100% RH
80% RH
60% RH
40% RH
20% RH
0% RH

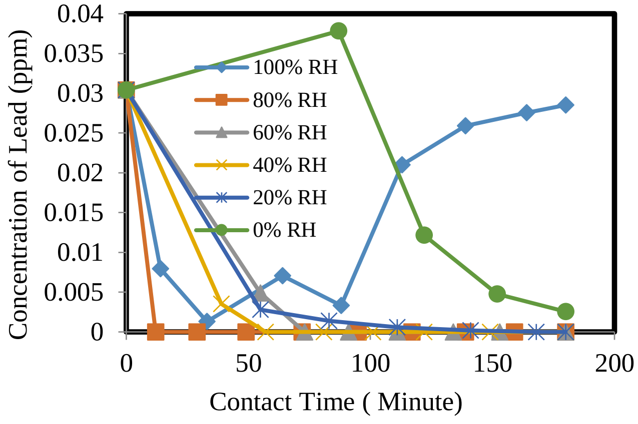
(g)


Concentration of Molybdenum(ppm)
Contact Time ( Minute)
0
2
4
6
8
10
12
14
0
50
100
150
200
100% RH
80% RH
60% RH
40% RH
20% RH
0% RH

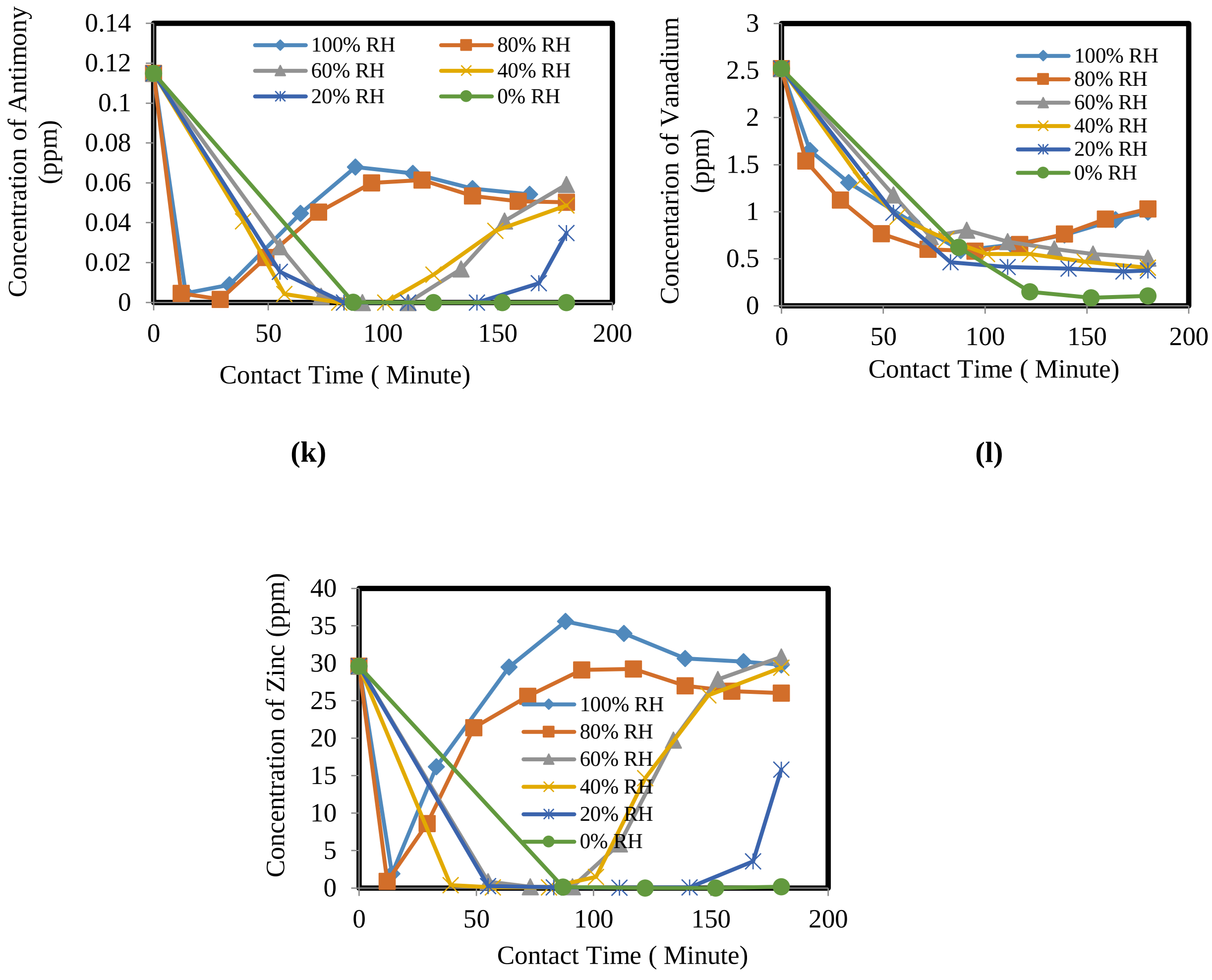
(h)


Concentration of Nickel (ppm)
Contact Time ( Minute)
0
0.01
0.02
0.03
0.04
0.05
0.06
0
50
100
150
200
100% RH
80% RH
60% RH
40% RH
20% RH
0% RH

(i)


Concentration of Lead (ppm)
Contact Time ( Minute)
0
0.005
0.01
0.015
0.02
0.025
0.03
0.035
0.04
0
50
100
150
200
100% RH
80% RH
60% RH
40% RH
20% RH
0% RH

(j)



**(k)** **(l)**

**(m)**

**Figure 6:** Adsorption of Heavy Metal Components by physically Treated Rice Husk and palm shell Hybrid Adsorbents.

## Conclusion

This study demonstrated the significant potential of physically treated hybrid adsorbents, combining rice husk and carbonized palm shell, for heavy metal removal from wastewater. Compared to single-component adsorbent (pure rice and pure carbonized palm shell), the hybrid material exhibited a notably enhanced adsorption capacity for cadmium, copper, vanadium, lead, antimony, and zinc in multi-layer bed column experiments. This improvement is evident from the lower area under the curve (AUC) values in the graphical results. However, it’s important to acknowledge the complexities involved in multi-component adsorption. The AUC pattern for each metal varied across different hybrid adsorbent mixing ratios, highlighting potential competition factors and roll-up effects. Interestingly, the study also revealed that the impact of multi-layer beds on adsorption capacity differed among hybrid adsorbents. While some showed an increase, others exhibited a decrease. These finding suggest the need for future research to optimize the mixing ratios of hybrid adsorbent. Techniques like basic linear programming can employed to identify the optimal ratio that maximizes the total heavy metal adsorption capacity. Overall, this study paves the way for the development of efficient and innovative adsorbents for wastewater treatment, contributing to the mitigation of heavy metal contamination.

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