



Phosphoric Acid-Activated Rice Husk Biochar for Enhanced Cadmium Adsorption

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Abstract

In farming areas, the amount of rice husks piled up is difficult to clean up and water filled with heavy metals can make the food unfit to be consumed. In this study, the effectiveness of phosphoric acid to activate rice husk biochar was improved, which makes it more efficient in removing Cd ions from wastewater. The raw rice husk was weighed and mixed with H₃PO₄ in 1:1 ratio and heated at 550 degrees Celsius. The process is called pyrolysis. Using phosphoric acid treatment, the surface area went up from 64.2 to 268.4 m²/g, and it also successfully added phosphorus groups to the surface. The adsorption process was investigated by performing batch adsorption experiments for various initial concentrations, pH and contact times. The highest level of Cd²⁺ that was absorbed was 42.51 mg per gram at pH 6.0, which is 8 times as much as the absorption of the unmodified biochar of 5.12 mg per gram. The reaction rate is very well described by the pseudo-second-order model ($R^2 = 0.998$), suggesting that chemisorption is the major process occurring during the reaction. Better correlation was obtained when the equilibrium data were plotted on the Langmuir isotherm ($R^2 = 0.996$), indicating that the adsorption occurred in a single layer on sites that were distributed uniformly. This process converts low-value farm waste into a solid that can absorb and so provides a means of utilizing resources and ensuring farm systems are safe.

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Keywords: Rice husk biochar, chemical activation, cadmium adsorption, agricultural waste valorization, wastewater remediation.

Introduction

Fast industrial growth, lots of farming work, and bad ways of dealing with waste from both farming and industry have led to a lot of heavy metal pollution in farmland and water systems around the world. Cadmium is one of the harmful heavy metal pollutants, which is highly toxic, persistent in the environment and is not biodegradable (Mohan et al., 2007; Natasha et al., 2022). Cadmium contamination in farming regions primarily occurs from industrial wastes, mining contamination of the water, and excessive use of low quality phosphate fertilizer. Cadmium is readily transported through soil and is absorbed by plants through the roots. This leads to cadmium building up in the food chain. Zheng et al. (2022) reports that prolonged exposure to cadmium in foods can cause severe health problems such as kidney damage, bone problems, and various types of cancer. The reduction in use of cadmium-containing water for irrigation of plants and in farm effluents is crucial to ensure the safety of food in the world and to safeguard human health. The approaches to remove heavy metals from water include traditional chemical methods to settle out the metals, ion exchange between materials, filtering the water using a membrane process, and using electrical means to remove the metals (Tan et al., 2015). These, however, suffer from high operating costs, high energy consumption, and emission of harmful wastes, which makes their application challenging in less developed countries where energy and other resources are limited. However, its use in adsorption has been proving to be a great alternative since it is simple, inexpensive and can be designed in different ways (Regmi et al., 2012). In recent years, agricultural waste based adsorbents have become the main interests of the studies, in line with the primary objectives of the circular bioeconomy and the concept of "waste as a useful resource" (Tomczyk et al., 2020; Yaashikaa et al., 2020).

Growing rice produces a lot of rice husks, which is a big kind of farm waste that piles up in processing areas in warm and temperate regions. Not taking good care of land, such as burning fields in open areas, causes a lot of air pollution in the region and also releases bad gases that help make global warming worse (Li et al., 2022). Changing agricultural waste into biochar using a method called oxygen-limited thermal pyrolysis is a good way to reuse waste in a friendly way for the environment (Qiu et al., 2021). Even though rice husk biochar is quite clean, it often has a small surface area and not many active sites on its surface, so it doesn't hold heavy metal ions very well (Zhang et al., 2020). To solve these issues, we need to apply particular surface engineering and chemical activation techniques that are aimed at the target.

Using phosphoric acid (H_3PO_4) to bring about chemical changes can be a good method to enhance biochar. Phosphoric acid is active simultaneously with another acid, which is used to remove water from biopolymers. It also provides a framework to reduce a too large amount of shrinking of the carbon material during heat treatment. This will result in a lot of small pores inside the material, both very small and slightly bigger ones (Liu et al., 2020). By activation, H_3PO_4 is able to incorporate stable oxygenated phosphanes directly into the carbon structure, for example in the form of phosphate esters. This process generates more active sites available on the surface to be attached to the heavy metals, which is reported in the study of Zhou et al. (2021). Despite the abundance of research on general acid treated biochars, few studies have focused on the optimal application of H_3PO_4 activated rice husk biochar for the removal of Cd^{2+} from water in agricultural environment, particularly under water scarcity.

This short communication explains a better way to make high-quality phosphoric acid-activated rice husk biochar (PA-RHB). We carefully check how its physical structure changes, how the surface chemistry develops, and how well it binds cadmium. This study uses kinetic and equilibrium isotherm models to explain the processes that help better trap cadmium, offering a useful and large-scale method to turn waste into valuable resources for cleaning polluted farm water.

Materials and Methods

Materials and Reagents

Raw rice husk was gathered from a nearby agro-processing milling plant located in Anand, Gujarat, India. The biomass was washed well with deionized water to remove dirt and dust, then dried in an oven set to $105^\circ C$ for 24 hours until it reached a constant weight, and finally ground using a machine until it passed through a 100-mesh sieve. All the chemical reagents used in this research, like phosphoric acid (H_3PO_4 , 85% w/w), cadmium nitrate tetrahydrate ($Cd(NO_3)_2 \cdot 4H_2O$), hydrochloric acid (HCl, 0.1 M), and sodium hydroxide (NaOH, 0.1 M), were of high quality and bought from Sigma-Aldrich. A stock solution of Cd^{2+} at 1000 mg per liter was made by dissolving the right amount of $Cd(NO_3)_2 \cdot 4H_2O$ in distilled water and then carefully diluting it step by step to create the needed working concentrations.

Preparation of Biochars

To make pristine rice husk biochar (R-RHB), the treated biomass was placed in a quartz tube furnace and heated to $550^\circ C$. The temperature was increased steadily at a rate of $10^\circ C$ per minute. Throughout the process, a steady flow of ultra-pure nitrogen gas was passed through at a rate of 200 mL per minute. Once the temperature reached $550^\circ C$, it was kept at that level for 2 hours. To make phosphoric acid-activated rice husk biochar (PA-RHB), dried rice husk was combined with an 85% H_3PO_4 solution in an exact 1:1 ratio by mass, where the dry rice husk was matched with the amount of pure H_3PO_4 . The slurry was stirred at $80^\circ C$ for 4 hours to make sure the acid fully soaked into the cell structure, then dried at $105^\circ C$ for 12 hours, and heated in the same way as before— $550^\circ C$ for 2 hours under nitrogen gas. After heating, both biochars were washed several times with 0.1 M HCl and then boiled deionized water until the liquid left after settling had a neutral pH, around 7.0, which removed any leftover acid and soluble minerals. The final biochars were dried at 80 degrees Celsius for 15 hours and kept in vacuum-sealed desiccators.

Characterization Protocols

The total volume of pores, its surface area, and mean size of the pores were measured using a nitrogen desorption and adsorption test at $-196^\circ C$ with a Micromeritics ASAP 2020 machine. The results were obtained using standard BET and BJH methods. Surface functional groups were studied with the help of Fourier-transform infrared spectroscopy (FTIR)

over a wavelength range from 4000 to 400 cm^{-1} with a Nicolet iS50 spectrometer. The elements like hydrogen, nitrogen, carbon, and sulfur were measured using an Elementar Vario EL Cube analyzer. The amount of oxygen was measured by the total mass of the sample and subtracting the combined mass of the other elements.

Batch Adsorption Experiments

Three groups of adsorption tests were carried out using three 100-mL Erlenmeyer flasks, each holding 50 mL of a solution that had Cd^{2+} ions in it. Unless we were looking at specific operational factors, the standard baseline conditions remained the same: 2.0 grams of adsorbent for each liter of solution, a starting pH of 6.0, a stirring speed of 150 revolutions per minute, and a temperature of 25 degrees Celsius. The effect of solution pH was looked at over a range from 2.0 to 7.0. Small amounts of 0.1 M HCl or 0.1 M NaOH were added to change the pH slightly. Kinetic studies were carried out at regular time intervals between 5 and 240 minutes, starting with an initial concentration of 50 mg/L of Cd^{2+} . Equilibrium isotherm studies were done by changing the starting cadmium concentration from 10 to 100 mg/L and letting the process run for 240 minutes to make sure that full thermodynamic equilibrium was reached. After the agitation process, the samples were passed through 0.45-micron membrane filters. The remaining amount of cadmium ions in the water was then measured using a technique called Atomic Absorption Spectroscopy, which was done with a Shimadzu AA-7000 device. The equilibrium adsorption capacity, which is measured in milligrams per gram (mg/g), was calculated using the mass balance equation: q_e equals $(C_0 \text{ minus } C_e)$ multiplied by V , then divided by M . Here, C_0 is the initial concentration of cadmium in the liquid phase (mg/L), C_e is the concentration after equilibrium is reached (mg/L), V is the volume of the solution (in liters), and M is the dry weight of the biochar used as the adsorbent (in grams).

Mathematical Modeling and Statistical Analysis

Adsorption kinetics were studied using two types of kinetic models: non-linear pseudo-first-order and pseudo-second-order (Fan et al., 2020). Adsorption equilibrium profiles were studied using the traditional Langmuir and Freundlich isotherm models (Li et al., 2021). The fit was checked using the R^2 value and the chi-square error. The experimental data were analyzed using a one-way ANOVA test through SPSS 25.0, and results were considered statistically significant if the p -value was less than 0.05.

Results and Discussion

Physicochemical and Structural Transformation

The surface texture and the amounts of different components in R-RHB and PA-RHB have changed clearly in structure due to the phosphoric acid treatment, as shown in Table 1. Using phosphoric acid to activate the material resulted in more biochar being produced, increasing from 38.4% to 44.1%. This indicates that phosphoric acid is effective as both a flame retardant and a chemical activator. By facilitating the formation of rings and preventing carbon-based materials from escaping into the atmosphere, it aids in the bonding of biopolymers (Qiu et al., 2021). The big decrease in ash content from 16.1% to 6.8% occurred because a strong acid treatment took out a lot of the acid-soluble inorganic minerals, mostly silica, from the raw rice husk during processing.

One of the biggest changes was that the BET surface area increased by over 300%, rising from 64.2 m^2/g in R-RHB to 268.4 m^2/g in PA-RHB. The structure is getting bigger, and at the same time, the total amount of pore space has almost doubled four times, increasing from 0.078 to 0.294 cm^3/g . The change happens because an acid breaks down cellulose, hemicellulose, and lignin, which takes away water from these materials. This reaction forms phosphate bonds that physically stretch the carbon structure. After the washing steps, these internal polyphosphate compounds are taken away, showing a big network of new small and medium pores (Hassan et al., 2020). The average pore size went down from 4.86 nm to 3.12 nm, which shows that there was a lot of growth in the smaller pores inside the char structure.

The elemental analysis shows that the carbon content rises from 58.32% to 69.15%, which means there is more aromatic condensation happening. The presence of a specific amount of phosphorus, 3.68%, in PA-RHB shows that stable phosphorus compounds are properly included in the carbon structure. FTIR analysis showed major changes in the surface chemistry. The R-RHB spectrum showed normal stretching vibrations for aliphatic -OH groups around 3410 cm^{-1} , C=O bonds in aromatic compounds around 1605 cm^{-1} , and asymmetric Si-O-Si bonds around 1080 cm^{-1} . On the other hand, the PA-RHB spectrum showed a clear drop in the Si-O-Si peak, along with the emergence of two separate absorption bands at 1165 cm^{-1} and 985 cm^{-1} . These bands show the stretching of P=O bonds and the stretching of P-O groups in phosphate ester structures, as found by Liu et al. in 2020. These changes show that using phosphoric acid to activate the biochar effectively adds long chains of polyphosphate and oxygen-containing phosphorus compounds to the inside surfaces of the biochar structure.

Critical Impact of Solution pH

The beginning pH level greatly affects the charge on the adsorbent's surface and how heavy metal ions act and change in water. The experimental results show how the pH level influences the ability of R-RHB and PA-RHB to remove Cd^{2+} ions, using data collected from pH levels between 2.0 and 7.0. At very acidic conditions, like a pH of 2.0, neither material absorbed a lot of cadmium. Their ability to take in cadmium was limited, with R-RHB able to take in up to 0.84 mg per gram and PA-RHB able to take in up to 4.21 mg per gram. This limited adsorption occurs mostly due to competition, where many hydronium (H^+) ions stick to functional groups such as carboxyl, hydroxyl, and phosphate, causing the surface to become positively charged. These positive charges then repel the positively charged Cd^{2+} ions through electrostatic forces, as reported by Li et al. in 2017.

When the solution's pH was increased from 3.0 to 6.0, both biochars had a significant improvement in their ability to absorb substances. The PA-RHB biochar continued to perform better than the raw biochar during this entire process. At pH 6.0, PA-RHB showed the highest stable adsorption capacity of 38.65 mg/g using the basic kinetic parameters, whereas R-RHB had a much lower capacity of only 4.88 mg/g. This clear improvement occurs because the surface groups gradually give off protons as the number of hydronium ions goes down. Removal of protons creates negative charges on the functional sites which increase attraction of Cd^{2+} ions by electrostatic attraction. Furthermore, the attached phosphate groups are not as likely to release protons as are the typical carboxyl and hydroxyl groups, thus the pH-dependent

functionality of the PA-RHB remains high across a broad pH range as described by Zhou et al. in 2021. Cadmium ions will start to react with the hydroxide ions and form a chemical compound known as $\text{Cd}(\text{OH})_2$ which is solid. This solid accumulates on the surface, and can appear as if more cadmium is being consumed than is actually. So, pH 6.0 was picked as the best starting point for all future experiments.

Adsorption Kinetics Modeling

The time course of cadmium bound to R-RHB and PA-RHB reveals the important events that slow the process down with time (5 to 240 minutes, see legend in Figure 1). Cadmium bonds to PA-RHB in two processes: immediately, in the first 30 minutes, since there are a lot of active sites on the surface. Over the next few hours, it slowly penetrates the inner part of the material, then the amount of cadmium bound begins to stabilise after 120 minutes, when the inner sites are full. Non-linear modelling showed that the pseudo-second-order kinetic model fit the PA-RHB data very well ($R^2 = 0.998$, $\chi^2 = 0.11$), which is much better than the pseudo-first-order model ($R^2 = 0.884$). This is a high match, indicating that chemisorption (chemical bonding and electron sharing between Cd^{2+} ions and the surrounding P and O groups) is the rate-limiting process (Zhang et al., 2021).

Equilibrium Isotherm Profiles and Comparative Analysis

Equilibrium isotherms were prepared by using different initial amounts of cadmium (see legend of figure 2), to obtain the maximum amount of heat stored in a single layer. This adsorption data is well described by Langmuir equation with high R^2 value of 0.996 as compared with Freundlich equation with R^2 value of 0.912. This reflects the uniform deposition of 1 layer on a common set of identical surface sites and the lack of any mutual interaction from the molecules between surface sites (Li et al., 2021). The maximum amount of materials that can be adsorbed in a single layer of PA-RHB is 42.51 mg/g which is 8.3 times higher than the amount of material adsorbed by the original R-RHB material which is 5.12 mg/g. To place these results in perspective, Table 2 presents the results of PA-RHB sorbent performance in comparison with other recently studied sorbents from the raw and modified agricultural residue category. The data in Table 2 shows that the rice husk biochar made in this study, which was activated with phosphoric acid, has a better ability to absorb cadmium compared to many other types of agricultural waste. Chemically altering the surface with KOH or K_2CO_3 may cause the internal pores to increase in size and alter the structure on the surface. The methods, however, do not introduce specific heavy metal ions which bind to specific targets. PA-RHB is highly effective due to two reasons: its large surface area (268.4 m^2/g) enables the substances to reach the surface easily, and a large number of phosphorous groups on the surface enable strong binding of metal cations with a charge of two. This was found by Cui et al. in 2021.

Elucidation of Sorption Mechanisms

Several physical and chemical processes occur simultaneously to make the big increase in cadmium adsorption capacity observed for PA-RHB.

1. The FTIR results indicate that there are numerous phosphate groups along the carbon structure. These sites contain oxygen which is used to form strong Lewis bases that are able to donate electron pairs to the Lewis acids Cd^{2+} ions. This interaction forms very stable and stable surface complexes (Liu et al., 2020).
2. Ion exchange is when the divalent cadmium ions are exchanged for protons or other mineral cations (such as calcium or potassium) bound to oxygen-containing structures present in the activated biochar. This process will generate hydronium ions (Li et al., 2017) in the solution.
3. At the optimum working pH of 6.0, the surface sites de-protonate, leaving a negative charge on the surface. This may facilitate the more direct and greater movement of the positive Cd^{2+} ions by electrostatic attraction towards the periphery of the biochar (Ghorbani et al., 2022).
4. Pore filling: The larger BET surface area and higher total pore volume (0.294 cm^3/g) results in a strong physical structure with a lot of capacity. This network facilitates the transport of a large amount of material, allowing hydrated cadmium complexes to pass through the very small pores within the structure, where they can be trapped and retained within the structure (Hassan et al., 2020).

Practical Implications for a Circular Bioeconomy

The conversion of agricultural wastes, such as rice husks, to particular environmental adsorbents by a single-step treatment with phosphoric acid is in line with the concept of waste to value and circular bioeconomy. The process transforms a waste material which is traditionally burned in open fields into a useful and valuable material. Agricultural by-product usage is a way to reduce raw material costs. The temperature for thermal activation of H_3PO_4 is significantly lower (550°C), as compared to the higher temperatures (above 800°C) required for traditional methods of thermal activation, involving the use of steam or carbon dioxide (Qiu et al., 2021). The biochar-treated water can be used to manage wastewater in agricultural economies; it is a low-cost solution to remove contaminated irrigation water and prevent accumulation of pollutants in food production.

Conclusions

The result of this research indicates that the application of phosphoric acid in a single process is a good method to produce an adsorbent material, which is capable of high cadmium adsorption, to remove the contamination. The specific surface area was raised from 64.2 to 268.4 m^2/g by treating them with phosphoric acid and it has introduced functional phosphorus complexes to the surface. The adsorption data revealed that Langmuir model is suitable and a maximum of 42.51 mg of cadmium could be adsorbed per gram at pH 6.0. This is 8 times more effective than the unmodified biochar. The kinetic profiles were best represented by the pseudo-second order model indicating that chemisorption of the reaction was the primary limiting factor. This process is based on the electrostatic attraction between particles and surface and the formation of surface complexes. These batch tests have good results in controlled environments, but further tests are warranted to determine the performance of the system in real applications such as continuous fixed-bed columns. Combining multi-metal agricultural effluents will be useful to determine whether this will be able to be reused and upscaled to larger applications. The resource valuation approach will help establish a circular economy and reduce the excess agricultural wastes and heavy metal contamination in the irrigation system.

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Table 1. Physicochemical and Structural Properties of Raw (R-RHB) and Phosphoric Acid-Activated (PA-RHB) Rice Husk Biochars

Physicochemical Property	R-RHB	PA-RHB
Biochar Yield (%)	38.4 ± 1.2	44.1 ± 0.9
Ash Content (%)	16.1 ± 0.5	6.8 ± 0.3
BET Specific Surface Area (m ² /g)	64.2 ± 2.4	268.4 ± 5.1
Total Pore Volume (cm ³ /g)	0.078 ± 0.004	0.294 ± 0.008
Average Pore Diameter (nm)	4.86 ± 0.15	3.12 ± 0.11
Elemental Composition (wt.%)		
Carbon (C)	58.32 ± 0.85	69.15 ± 0.72
Hydrogen (H)	2.11 ± 0.06	1.45 ± 0.04
Nitrogen (N)	0.45 ± 0.02	0.31 ± 0.01

Oxygen (O)*	23.02 ± 0.64	22.29 ± 0.55
Phosphorus (P)	< 0.01	3.68 ± 0.12

*Calculated by mass difference.

Table 2. Comparison of Maximum Monolayer Cadmium (Cd^{2+}) Adsorption Capacities of Various Agricultural Biochar Materials

Biomass Feedstock Source	Activation/Modification Agent	q_{max} (mg/g)	Reference
Switchgrass Residues	Hydrothermal Carbonization	4.12	Regmi et al., 2012
Pecan Shells	Direct Pyrolysis	5.23	Mohan et al., 2007
Dairy Manure Digestate	Standard Slow Pyrolysis	11.24	Ahmad et al., 2014
Sugarcane Bagasse	KOH Activation	28.45	Penido et al., 2019
Broad Bean Stems	K_2CO_3 Modification	31.20	Ghorbani et al., 2022
Rice Husk Byproduct	Phosphoric Acid (H_3PO_4)	42.51	This Study

Fig. 1 Adsorption kinetics of cadmium onto R-RHB and PA-RHB

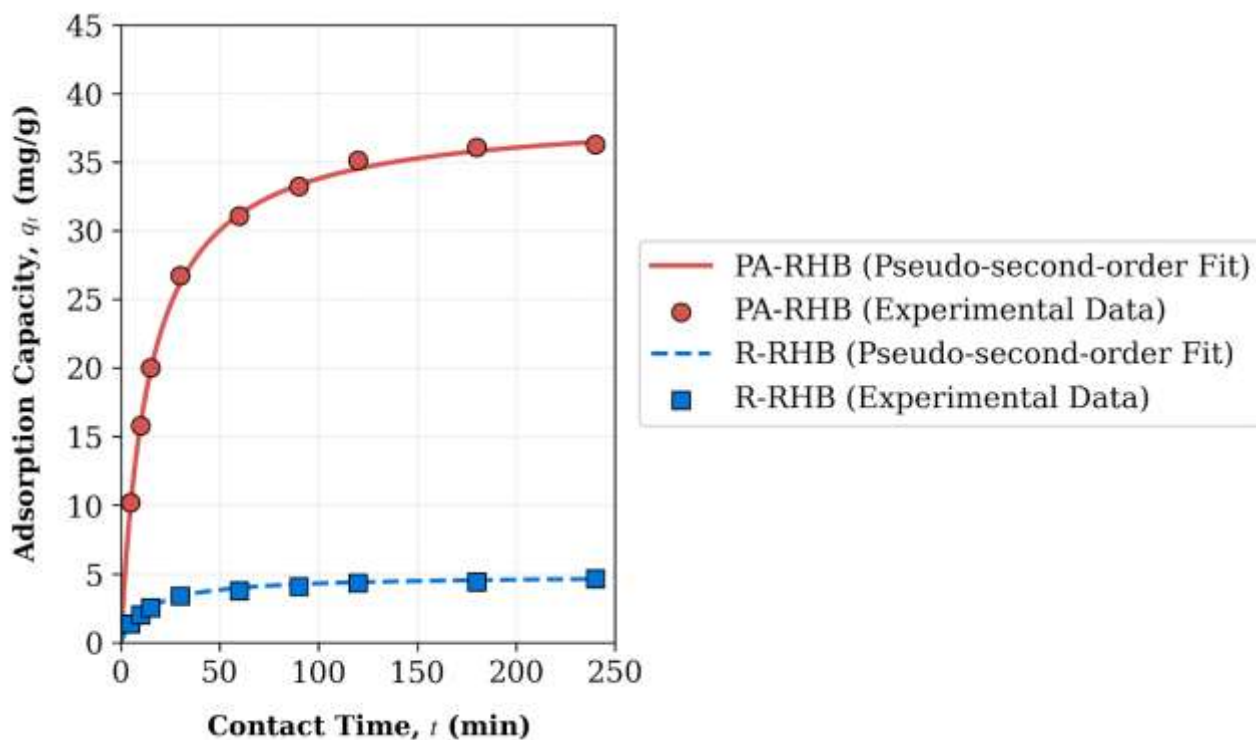


Fig. 2 Adsorption isotherms of cadmium onto R-RHB and PA-RHB at 25°C

