University of Tehran Press

Pollution

https://jpoll.ut.ac.ir/

Print ISSN: 2383-451X
Online ISSN: 2383-4501

Biochar Derived from the Husk and Straw of Rice (*Oryza sativa* L.) Produced via Low-Temperature Pyrolysis as an Effective Adsorbent for Pb (II) Removal

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Article Info

Article type:
Research Article

Article history:
Received: 26 Apr 2023
Revised: 22 Jun 2023
Accepted: 16 Sep 2023

Keywords:
Biochar
Biomass
Biosorption
Lead removal
Rice straw

ABSTRACT

Pyrolysis is a promising thermochemical conversion process that transforms biomass into biochar, a carbon-rich solid material, in an oxygen-limited environment. This study focuses on the utilization of rice byproducts, namely rice straw and rice husk as feedstock for biochar production through low-temperature pyrolysis. The aim is to explore the potential of these biochars as cost-effective adsorbents for removing metal contaminants from aqueous solutions, with a particular emphasis on Pb(II) removal. Physicochemical properties of the biochars produced at a low temperature of 300 °C were thoroughly investigated, including surface morphology and their adsorption capacity for Pb(II). Remarkably, the rice straw biochar (RSB) produced at 300 °C exhibited exceptional Pb(II) adsorption capacity, with a value of 390.10±0.30 mg/g, and demonstrated a high Pb(II) removal efficiency of 96.10±0.30% when modified with 30% w/w H2O2. A crucial aspect of this study lies in the evaluation of the cost-effectiveness of the biochar production process, particularly when compared to commercially available adsorbents. By demonstrating the potential of rice byproduct-derived biochar as an efficient Pb(II) biosorbent in aqueous environments, this work not only provides new insights into the preparation of biochar using low-temperature pyrolysis but also offers a viable and economical solution for metal-contaminated water treatment. The findings of this research contribute to the field of sustainable waste utilization and highlight the significant potential of rice byproduct-based biochar as an environmentally friendly adsorbent for heavy metal removal.

Cite this article: Chaijak, P., Michu, P., Thipraksa, J., & Kongthong, A. (2023). Biochar Derived from the Husk and Straw of Rice (Oryza sativa L.) Produced via Low-Temperature Pyrolysis as an Effective Adsorbent for Pb (II) Removal. *Pollution* , 9 (4), 1666-1675.
https://doi.org/10.22059/POLL.2023.358321.1888

DOI: https://doi.org/10.22059/POLL.2023.358321.1888

INTRODUCTION

In recent years, the release of heavy metals into the environment has been on the rise due to various anthropogenic activities, including industrial processes, energy consumption, mining operations, and the use of fertilizers and pesticides (Ihsanullah et al., 2016; Sankhla et al., 2016; Siddiqui and Pandey, 2019). Among these heavy metals, lead (Pb) poses a significant risk due to its toxic nature, multiple sources, and resistance to biodegradation, leading to an increase in its concentration in wastewater and becoming a matter of serious environmental concern (Zhou et al., 2017).

To address this issue, several techniques such as chemical precipitation, ionic exchange, reverse osmosis, and adsorption have been employed, with adsorption being considered the

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most promising method due to its high efficiency and operational simplicity (Li et al., 2016). However, the widespread use of adsorption is hindered by the lack of economically viable and environmentally friendly adsorbents, necessitating the development of sustainable and low-cost alternatives for effective heavy metal remediation (Park et al., 2015).

Biochar, a porous and finely-textured material produced through slow pyrolysis in a low-oxygen environment at temperatures below 900 °C, has emerged as a potential solution. It can be derived from various sources such as forestry and agricultural waste, sawdust, algae, and poultry manure (Liu et al., 2015; Abdul et al., 2017). Biochar possesses a large surface area, porous structure, abundant surface functional groups, and high cation exchange capacity (CEC), making it a promising adsorbent for pollutants (Chandraiah, 2016). Compared to activated biochar and carbon, unmodified biochar is less expensive as it does not require an additional activation process. However, there have been attempts to enhance its adsorption capacity through biochar modification, which can result in waste generation, increased costs, and potential environmental pollution from the use of chemical reagents (Wang et al., 2018; Singh et al., 2019; Niazi et al., 2018). Therefore, identifying unmodified biochar with high heavy metal adsorption capacity is crucial. The physicochemical properties and heavy metal adsorption capacity of biochar are known to be significantly influenced by the raw materials used. Hence, the selection of appropriate feedstock plays a vital role in developing efficient biochar adsorbents (Suman et al., 2017; Zahedifar, 2017).

Rice being a staple food in many Asian regions, including China, India, Indonesia, Bangladesh, Vietnam, Thailand, Myanmar, Japan, the Philippines, South Korea, and Pakistan, generates a substantial amount of rice production residue during the milling process. Annually, Asia produces approximately 600-800 million tons of rice straw, while global production ranges from 800-1000 million tons (Karam et al., 2022). Rice husk and rice bran also contribute to the overall amount of rice production residue, with annual production levels of 120 tons and 76 million tons, respectively (Bodie et al., 2019). Considering the abundance of rice production residue, utilizing it as a feedstock for biochar production offers a promising opportunity to develop effective and sustainable adsorbents for heavy metal remediation. This study aims to investigate the potential of unmodified rice-based biochar as an adsorbent for lead (Pb) removal from wastewater, focusing on its adsorption capacity, optimization of process parameters, and evaluation of its efficiency in real-world applications.

The utilization of biochar for wastewater treatment has gained significant attention in recent years due to its low-cost, eco-friendly, and effective adsorption properties. In this study, researchers focused on preparing and characterizing biochar from rice byproducts such as rice husk and rice straw for the removal of Pb (II) from wastewater. The study had 2 specific objectives. Firstly, to prepare and characterize the biochar from rice byproducts and secondly, and to assess their adsorption capacity for Pb (II) in wastewater. The research aimed to provide technical support for the utilization of rice byproduct biochar and the removal of Pb (II).

MATERIALS & METHODS

Materials

The byproducts of rice (*Oryza sativa* L.) were sourced from a local paddy field in Phatthalung province, Thailand, specifically the rice husk and rice straw. The properties of the rice husk and rice straw utilized in the experiment are summarized in Table 1. To prepare the feedstock for biochar production, the rice husk and rice straw underwent a drying process at 60 °C for 48 hr, ensuring their moisture content was sufficiently reduced.

Biochar preparation and surface morphology

The rice husk and rice straw were initially ground and sieved using a 40-mesh sieve to ensure a uniform particle size. Subsequently, the ground materials were subjected to carbonization in

Table 1. Properties of rice husk and rice straw used in this experiment.

Composition	Rice husk	Rice straw
Cellulose (% w/w)	30.01±0.23	34.10±0.22
Hemicellulose (% w/w)	23.20±0.10	20.40±0.15
Lignin (% w/w)	20.50±0.25	14.50±0.10
Ash (% w/w)	17.00±0.10	19.50±0.23

a muffle furnace under an oxygen-limited atmosphere. The carbonization process was carried out at a temperature of 300 °C for a duration of 2 hr. After carbonization, the resulting rice husk biochar (RHB) and rice straw biochar (RSB) were allowed to cool down before further use in the experiments. To enhance the surface properties of the biochar samples, gold (Au) and palladium (Pd) particles were deposited onto the biochar surfaces using an Ion Sputter Coater (SC7620, Quorum). The coated biochar samples were then analyzed using scanning electron microscopy (Quanta 450, FEI) to visualize the surface morphology and distribution of the deposited particles. The scanning electron microscopy analysis provided detailed information about the surface characteristics, topography, and particle distribution of the modified biochar samples. This characterization step was crucial for understanding the physical and chemical properties of the biochar, which influenced its adsorption capacity and effectiveness for heavy metal remediation.

Preparation of Pb(II) stock solution

A stock solution of Pb(II) with a concentration of 1000 mg/L was prepared by dissolving 1.599 g of $Pb(NO_3)2$ in 1000 mL of deionized water, following the method described by Wu et al. (2019). This stock solution was then further diluted to obtain a 200 mg/L Pb(II) solution for use in the adsorption experiments.

Adsorption experiment

For the adsorption experiments, various doses of biochar (0.1, 0.5, 1.0, 2.0, and 5.0 g/L) were added to 50 mL of the 200 mg/L Pb(II) solution. The mixture was agitated at 150 rpm and room temperature for 60 min. Additionally, to investigate the influence of different initial Pb(II) concentrations, 0.1 g of biochar was added to 50 mL of Pb(II) solutions with concentrations of 50, 100, 150, 200, 300, and 400 mg/L. The experiments were conducted at room temperature, with agitation at 150 rpm for a duration of 60 min.

Measurement of Pb(II) concentration

To determine the Pb(II) concentration in the solution after the adsorption experiments, the solution was filtered through filter paper. The filtered solution was then subjected to measurement using the 2,5-dimercapto-1,3,4-thiadiazole (DMTD) method, as described by Ahmed and Mamun (2001). In brief, 1 mL of the sample was mixed with 1 mL of DMTD solution (4.4×10^{-3} M), followed by the addition of 2 mL of 0.005 M HCl. The mixture was incubated for 1 minute before measuring the absorbance at 375 nm using UV-Vis spectrophotometry. The Pb(II) concentrations were confirmed using atomic absorption spectroscopy for validation and accuracy.

Calculation of Pb(II) removal

The percentage of Pb(II) removal was determined using the following equation (Eq. 1):

$$\text{Pb (II) Removal (\%)} = [(C_0 - C_t) / C_0] \times 100 \qquad (1)$$

where C_0 is the initial concentration of Pb(II) in the solution, and C_t is the concentration of Pb(II) in the solution after the adsorption process.

Biochar activation

For the activation of biochar, 5 g of the biochar sample was added to 50 mL of H_2O_2 solution with varying concentrations of 10%, 20%, and 30% (w/w). The mixture was allowed to soak for a duration of 2 hr. After soaking, the biochar was thoroughly washed with deionized water and subsequently dried at 80 °C overnight. This activation process aimed to enhance the adsorption properties of the biochar for subsequent Pb(II) removal experiments.

Pb(II) removal experiment

The Pb(II) removal experiment was conducted using 1 g of the activated biochar. Initially, 50 mL of Pb(II) solution with an initial concentration of 200 mg/L was prepared. The activated biochar was added to the Pb(II) solution, and the mixture was agitated for a specific duration to ensure equilibrium. The equilibrium Pb(II) concentration (CE) was determined after reaching equilibrium.

Calculation of adsorption capacity

The adsorption capacity (Qe) of the biochar was calculated using the following equation (Eq. 2):

$$Qe\ (mg/g) = (C_A - C_E) \times (V/M) \tag{2}$$

where Qe represents the adsorption capacity of the biochar in mg/g, C_A represents the initial concentration of Pb(II) in the solution, C_E represents the equilibrium concentration of Pb(II) in the solution, V represents the volume of the Pb(II) solution, and M represents the mass of the biochar used.

Effect of pH and contact time

Based on the methodology described by Han et al. (2017), the effect of pH and contact time on the experiment was investigated. The pH range tested was between 1.5 and 5.5, encompassing a variety of acidity levels. Four different contact times were employed, specifically 1, 8, 16 and 24 hr, allowing for a comprehensive assessment of the impact of duration on the experimental outcomes.

RESULTS & DISCUSSION

Enhancement of biomass properties through thermal processes

Thermal processes, such as hydrothermal carbonization (HTC) and low-temperature pyrolysis (LTP), have emerged as effective methods for improving biomass properties and producing densified biochar (Saveyn et al., 2009). Among these techniques, low-temperature pyrolysis (LTP) is particularly advantageous for biomass upgrading, as it can convert volatile matter into tar or non-condensable gases at lower temperatures (200-500 °C) under a reducing atmosphere (Abdullah and Wu, 2009). In this study, the byproducts of rice husk and rice straw were obtained after undergoing a low-temperature pyrolysis process at 300 °C for 2 hr. The resulting biochar samples were further characterized and analyzed using scanning electron microscopy (SEM). The morphology of rice husk biochar (RSH) and rice straw biochar (RSB) is presented in Figure 1 and Figure 2, respectively.

To evaluate the influence of biochar concentration on Pb(II) removal efficiency, varying doses of biochar were employed. Figure 3 depicts the Pb(II) removal efficiency of biochar at different concentrations. The highest Pb(II) removal, with an efficiency of 85.10±0.10%, was

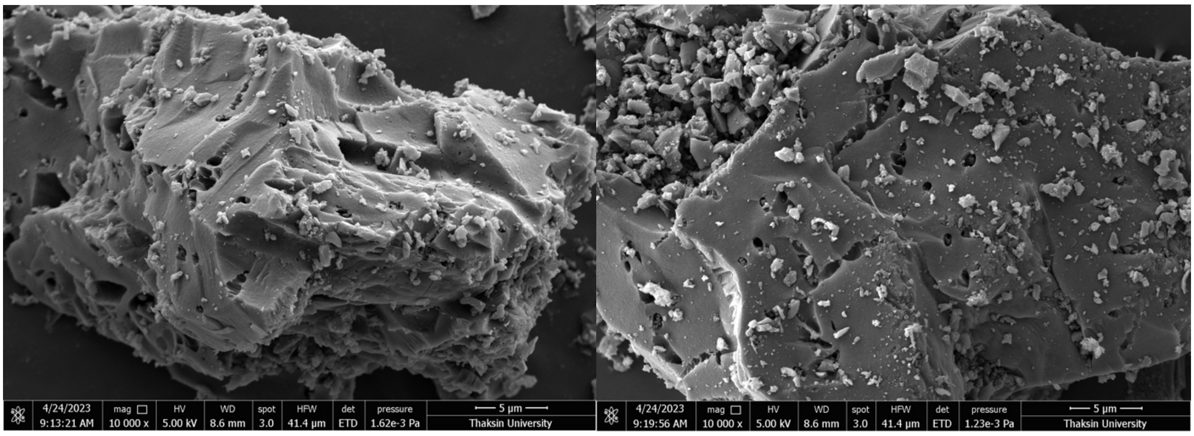

Fig. 1. The surface morphology of the RHB under SEM.

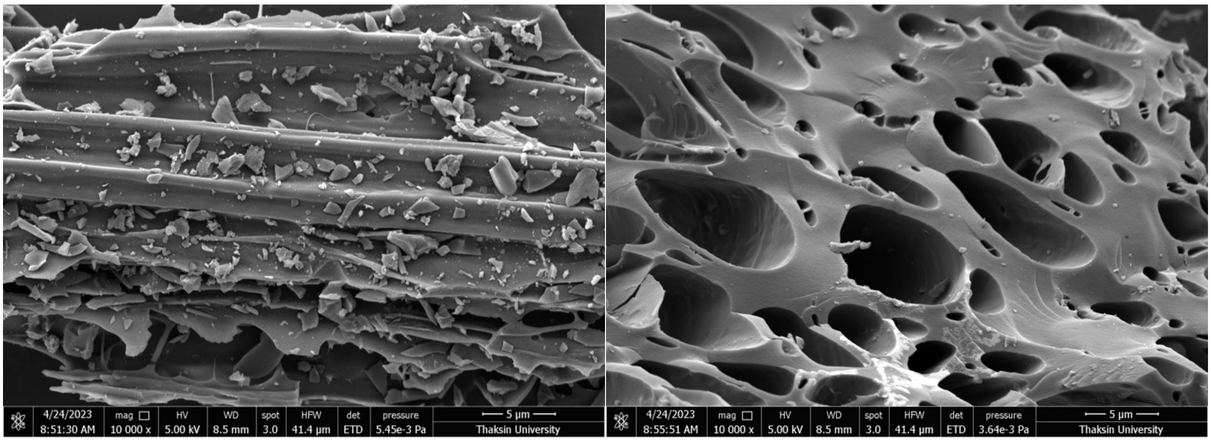

Fig. 2. The surface morphology of the RSB under SEM.

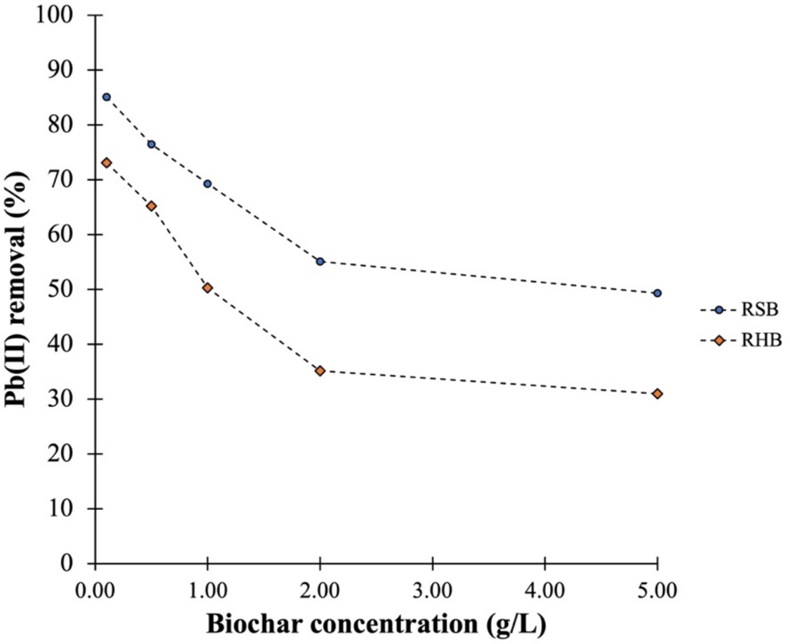

Fig. 3. The effect of the adsorbent concentration on Pb(II) removal

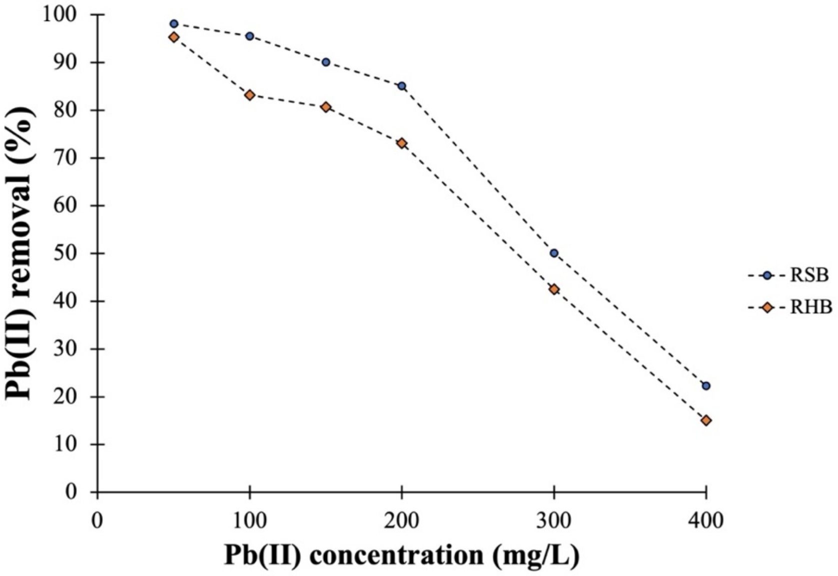

Fig. 4. The effect of the Pb(II) concentration on Pb(II) removal.

achieved using 0.1 g/L of RSB, while 0.1 g/L of RHB exhibited a maximum Pb(II) removal efficiency of 73.10±0.01%. These results indicate that both RSB and RHB possess significant capabilities for Pb(II) removal.

Figure 4 illustrates the impact of Pb(II) concentration on the adsorption capacities of RSB and RHB. Notably, both biochar samples demonstrated exceptional adsorption capabilities at low doses. However, as the concentration of the adsorbent dose increased, the adsorption capacities of RSB and RHB decreased. This observation aligns with the findings reported by Zhou et al. (2017). The decrease in adsorption capacity with increasing adsorbent dose and fixed Pb(II) concentration can be attributed to the limited movement of Pb(II) ions and reduced solution volume, leading to saturation and aggregation of active sites on the biochar surface. These factors ultimately result in a decrease in the overall adsorption capacity (Akram et al., 2017).

Overall, the results indicate that the thermal treatment of rice husk and rice straw at 300 °C for 2 hr successfully produced biochar samples (RSH and RSB) with distinct morphological characteristics. Furthermore, the adsorption experiments demonstrated the significant potential of both RSB and RHB for Pb(II) removal, with optimal results achieved at specific biochar concentrations. These findings contribute to the understanding of biochar properties and their application in heavy metal remediation, highlighting the importance of biochar as a sustainable and effective adsorbent in environmental pollution control.

The RSB and RHB were modified by immersion in different concentrations of a peroxide agent. The Pb(II) removal efficiency of the modified biochar is presented in Figure 5. The optimization study revealed that the maximum removal of Pb was achieved at pH 5.0. At this pH level, the adsorption process exhibited its highest efficiency in removing Pb from the wastewater samples. Additionally, it was observed that complete removal of Pb was achieved within a contact time of 6 hr. This indicates that extending the contact time beyond 6 hr did not contribute to further improvements in Pb removal efficiency. These findings provide valuable insights for designing efficient Pb removal strategies, suggesting that pH 5.0 and a contact time of 6 hr are optimal conditions for achieving effective and rapid removal of Pb from wastewater. The highest Pb(II) removal efficiency was observed for the 30% w/w H_2O_2-modified RSB, which was 96.10±0.30% (adsorption capacity was 390.10±0.30 mg/g). While, the RHB showed

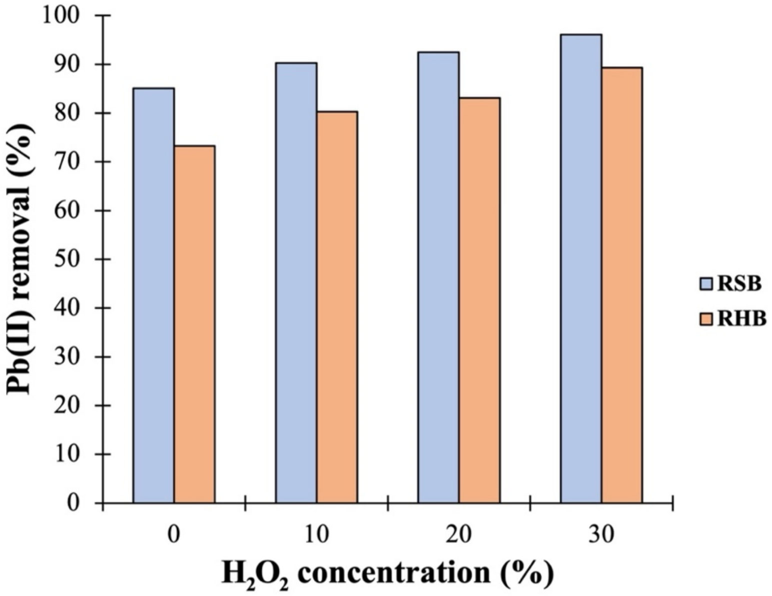

Fig. 5. The effect of H_2O_2 concentration on Pb(II) removal.

Table 2. The potential of rice byproducts for Pb(II) removal in aqueous phase

Rice byproducts	Pyrolysis temperature (°C)	Adsorption capacity (mg/g)	Reference
Rice straw	300	390.10±0.30	This study
Rice husk	350-450	17.6	Wijeyawardana et al. (2022)
Rice straw	700	222.6	Xu et al. (2022)
Rice straw	500	276.3	Li et al. (2018)
Rice husk	400	22.1	Han et al. (2017)

a maximal Pb(II) removal of 89.35±0.32%.

Several studies have investigated the potential of different rice byproducts for Pb(II) removal in aqueous solutions. Jin et al. (2020) examined the use of grape pomace pyrolyzed at 700 °C and achieved a maximum Pb(II) removal efficiency of 66.50% at an initial concentration of 300 mg/L. In a separate study, Sakhiya et al. (2022) investigated rice straw biochar produced at 500 °C and reported a maximum adsorption capacity of 17.93 mg/g within a pH range of 8.23 to 10.25. In contrast, Sun et al. (2019) synthesized rice husk biochar by incorporating $Fe(NO_3)_3$ and treating it with a $KMnO_4$ agent, resulting in a remarkable Pb(II) adsorption capacity of 148 mg/g.

Table 2 provides a comparison of the Pb(II) removal potential of different rice byproducts in the aqueous phase. It is evident that the various rice byproducts exhibit distinct capabilities for Pb(II) adsorption, highlighting the influence of the pyrolysis conditions, surface modifications, and the inherent characteristics of the biochar material. These findings underscore the importance of selecting appropriate rice byproducts and optimizing the pyrolysis or modification processes to enhance the adsorption capacity for effective heavy metal removal. Overall, the studies mentioned demonstrate the potential of rice byproducts, including grape pomace, rice straw biochar, and rice husk biochar, as viable options for Pb(II) removal. The diverse range of Pb(II) removal efficiencies and adsorption capacities observed in these studies emphasizes the need for further exploration and optimization of biochar production techniques to develop efficient and sustainable solutions for heavy metal remediation in aqueous environments.

CONCLUSION

In conclusion, this study explored the utilization of rice byproducts, specifically rice straw and rice husk, as feedstock for biochar production through low-temperature pyrolysis. The focus was on assessing the potential of these biochars as cost-effective adsorbents for removing metal contaminants, with a specific emphasis on Pb(II) removal. The biochars produced at a low temperature of 300 °C exhibited favorable physicochemical properties, including surface morphology, and demonstrated significant adsorption capacity for Pb(II). Notably, the rice straw biochar (RSB) produced at 300 °C showed exceptional Pb(II) adsorption capacity, reaching 390.10±0.30 mg/g, and exhibited a high Pb(II) removal efficiency of 96.10±0.30% when modified with 30% w/w H_2O_2. The cost-effectiveness of the biochar production process was also evaluated, highlighting its potential as an economically viable alternative to commercially available adsorbents.

This research contributes valuable insights into the preparation of biochar using low-temperature pyrolysis and demonstrates the significant potential of rice byproduct-derived biochar as an environmentally friendly adsorbent for heavy metal removal in aqueous environments. The findings underscore the importance of sustainable waste utilization and offer a promising solution for metal-contaminated water treatment. Overall, this study highlights the promising prospects of rice byproduct-based biochar as an efficient and cost-effective adsorbent for heavy metal remediation. It not only contributes to the field of sustainable waste management but also emphasizes the potential of biochar as an environmentally friendly solution for addressing water pollution challenges caused by heavy metal contaminants

ACKNOWLEDGEMENT

This work was supported by Thailand Science Research and Inno Office of National Higher Education Science Research and Innovation Policy Council, Thaksin University (research project grant) Fiscal Year 2023.

GRANT SUPPORT DETAILS

The present research has been financially supported by Thailand Science Research and Inno Office of National Higher Education Science Research and Innovation Policy Council, Thaksin University (research project grant) Fiscal Year 2023.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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