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Adsorptive Removal of Zn (II) from Aqueous Solutions by Using Chemically Modified and Unmodified Rice Husk ASH

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Abstract:
The contamination of aquatic environments by heavy metals, particularly Zinc (II), presents a significant challenge for industrial wastewater management due to its non-biodegradability and bioaccumulative nature. This study investigates the comparative adsorptive performance of unmodified and chemically modified Rice Husk Ash (RHA) for the removal of Zn (II) from aqueous solutions. RHA was synthesized via controlled combustion at 600°C and subjected to alkaline (NaOH) activation to enhance its porosity and surface functional group density, specifically increasing silanol (Si–OH) sites. Experimental investigations focused on the influence of critical parameters, most notably solution pH, which was evaluated across a range of 5.5 to 8.5. Results indicate that pH is the primary determinant of adsorption efficiency, with removal percentages significantly increasing as pH rises toward the neutral-alkaline range. At an optimal pH of 7.5, modified RHA achieved a removal efficiency of 77.65%, substantially outperforming the 60.95% removal rate observed for unmodified RHA. This enhancement is attributed to the increased surface area and optimized binding potential of the activated ash.
Kinetic data were modelled to understand the mechanism of uptake, with findings confirming that the adsorption process follows first-order reversible kinetics. The determination of the overall rate constant (K_1) through linearized plots (1-u_t versus t) further validates that chemical modification accelerates the removal rate compared to raw RHA. Thermodynamic and kinetic analyses support the conclusion that chemically modified RHA serves as an efficient, low-cost, and sustainable biosorbent. Valorising agricultural waste into an engineered, high-performance medium, this research demonstrates that surface modification provides a scalable and environmentally responsible strategy for mitigating Zn (II) contamination, aligning with the principles of a circular economy. The findings underscore the potential of RHA-based remediation to provide an effective, green engineering solution for industrial wastewater treatment.

Keywords: *Rice Husk Ash (RHA), Chemical Modification, Adsorption Isotherms, Kinetics, Wastewater Treatment.*

Introduction:

The rapid pace of global industrialization has brought significant advancements in technology and manufacturing, but it has also introduced a critical environmental challenge: the discharge of toxic heavy metals into aquatic ecosystems. Among the various pollutants, heavy metals are of particular concern due to their non-biodegradability, persistence in nature, and tendency to bioaccumulate within the food chain. Metals such as lead, cadmium, mercury, and zinc are frequently detected in industrial effluents originating from electroplating, mining, metallurgy, battery manufacturing, and plastic production. Once released into water bodies, these pollutants pose severe threats to both human health and ecological stability, often resulting in complex health complications, including neurological disorders, metabolic disruption, and organ damage. Ahmad, R., & Kumar, R. (2010)., Bhatnagar, A., & Sillanpää, M. (2010), Hadi, M., Samarghandi, M. R., & McKay, G. (2010)., Ho, Y. S., & McKay, G. (2000).

Zn (II) is an essential trace element required for various biological functions in humans, animals, and plants. However, when anthropogenic activities lead to concentrations that exceed regulated safety limits, it shifts from a nutrient to a toxic pollutant. Elevated levels of Zn (II) in wastewater can inhibit biological wastewater treatment processes, complicate downstream industrial operations such as membrane fouling or boiler scaling-and threaten aquatic biodiversity. Consequently, regulatory bodies, such as the U.S. Environmental Protection Agency (EPA), have designated zinc as a priority pollutant, mandating the treatment of industrial wastewater to minimize its environmental footprint before discharge. Kalderis, D., Kotti, M., Méndez, A., & Gascó, G. (2008).

To address this, various conventional remediation techniques have been employed, including chemical precipitation, ion exchange, membrane filtration, and reverse osmosis. While these methods are effective under certain conditions, they are often constrained by high operational costs, energy requirements, and the generation of secondary waste, such as toxic sludge. These limitations have catalysed a shift toward the development of more sustainable and economically viable alternatives, specifically focusing on biosorption. Biosorption utilizes biological materials to capture metal ions, offering a promising, eco-friendly solution for wastewater purification. Kumar, A., & Prasad, B. (2013)., Kyzas, G. Z., & Matis, K. A. (2015). , Li, Y., Wang, Q., Wang, Y., & Chen, J. (2011).

Rice husk, an abundant agricultural by-product of the rice milling industry, has emerged as a highly promising candidate for this purpose. Large quantities of rice husks are generated annually, often leading to disposal challenges such as open-field burning, which contributes to air pollution. When processed under controlled combustion, these husks produce rice husk ash (RHA)-a material rich in silica and possessing high porosity, which inherently provides a favourable structure for the adsorption of metallic species. The presence of diverse functional groups, such as hydroxyl, carboxyl, and silanol (Si-OH) groups on the RHA surface, facilitates the binding of metal ions through mechanisms like ion exchange, surface complexation, and electrostatic attraction. Mahvi, A. H., Maleki, A., & Eslami, A. (2004).,

Despite the effectiveness of natural RHA, research has increasingly focused on chemical modification to further enhance its adsorption capacity. Treatment with agents such as sodium hydroxide (NaOH), sulfuric acid (H_2SO_4), or hydrochloric acid (HCl) alters the surface chemistry of the ash, effectively increasing the density of active binding sites and improving surface area. Studies indicate that these modifications significantly reduce the time required to reach equilibrium and heighten the percentage of Zn (II) removal compared to unmodified samples. Malik, P. K. (2004).

Understanding the mechanics of this process including the influence of solution pH, initial metal concentration, contact time, and temperature is essential for optimizing RHA-based remediation systems. Furthermore, thermodynamic and kinetic modelling, such as the Langmuir and Freundlich isotherms, provide critical insights into the nature of the adsorption process, confirming whether the interaction is spontaneous, endothermic, or exothermic. By integrating waste management with environmental remediation, the use of chemically modified and unmodified rice husk ash represents a vital step toward a circular economy, offering a low-cost, scalable, and sustainable strategy to mitigate Zn (II) contamination in industrial wastewater.

The discharge of heavy metals from industrial sectors including electroplating, metallurgical processing, and battery manufacturing remains a global environmental crisis. Zinc (Zn (II)), while essential for biological systems at trace levels, becomes significantly toxic when released into aquatic ecosystems at elevated concentrations. Anthropogenic activities consistently introduce Zn (II) into wastewater, where it evades natural degradation, persists in sediments, and bioaccumulates throughout the trophic levels. The long-term presence of zinc in water sources disrupts aquatic biodiversity and poses severe risks to human health, necessitating stringent regulatory oversight and robust remediation strategies. Malkoc, E., & Nuhoglu, Y. (2007).

Conventional technologies utilized to mitigate this contamination, such as chemical precipitation, ion exchange, and membrane filtration, frequently encounter significant operational bottlenecks. Chemical precipitation often necessitates the addition of bulk reagents, which results in the generation of large volumes of hazardous chemical sludge, thereby creating secondary disposal challenges. Similarly, advanced methods like reverse osmosis and electrochemical treatment are often hindered by high capital costs, energy-intensive requirements, and a lack of suitability for treating dilute, high-volume wastewater streams. These limitations highlight an urgent demand for "green" engineering solutions that balance high removal efficiency with economic and environmental sustainability., Ncibi, M. C., Mahjoub, B., & Seffen, M. (2007).

Rice Husk Ash (RHA) has emerged as a premier candidate for low-cost, sustainable biosorption due to its unique chemical and physical properties. Produced as a byproduct of the rice milling industry, RHA is primarily composed of amorphous silica, providing a highly porous architecture with a substantial surface area. The inherent presence of reactive surface functional groups including silanol (Si-OH), carboxyl, and hydroxyl groups provides multiple active sites capable of sequestering divalent metal ions like Zn (II) through mechanisms such as ion exchange, electrostatic attraction, and surface complexation.

Beyond its technical suitability, the use of RHA aligns with the principles of a circular economy. By transforming an agricultural waste product that is typically subject to open-air burning into a valuable adsorbent, this approach effectively closes the waste-cycle loop. Furthermore, the ability to chemically modify the RHA surface to enhance its binding capacity transforms a passive waste material into a tailored, high-performance remediation tool, offering a practical and scalable solution for industrial wastewater treatment.

Experimental Methods: Preparation and Batch Adsorption Studies:

The experimental protocol for investigating the removal of Zn (II) using rice husk ash (RHA) involves three distinct phases: the preparation of the adsorbent, the chemical modification process, and the batch adsorption experiments.

1. Adsorbent Preparation and Modification:

Raw rice husks are first washed thoroughly with deionized water to remove surface impurities, dust, and organic debris. The cleaned husks are dried at 105°C for 24 hours. The dried biomass is then subjected to controlled combustion in a muffle furnace at 600°C for 4 hours to produce RHA. The resulting ash is sieved to a uniform particle size (e.g., 75-150 μm) to ensure consistent surface area exposure. For the chemically modified RHA, a portion of the ash is treated with an activating agent such as 0.1 M NaOH or HCl at a solid-to-liquid ratio of 1:20. The suspension is stirred for 2 hours at room temperature to facilitate the functionalization of the surface. Following treatment, the modified RHA is filtered, washed repeatedly with deionized water until the filtrate reaches a neutral pH, and finally dried in a vacuum oven at 80°C. This process increases the porosity and introduces a higher density of active binding sites (e.g., silanol groups) compared to the unmodified ash.

2. Batch Adsorption Experiments:

The batch adsorption studies are conducted to evaluate the equilibrium uptake of Zn (II) under various operating conditions. A series of 250 mL conical flasks containing a fixed volume of Zn (II) solution (prepared by dissolving analytical grade $ZnSO_4 \cdot 7H_2O$ in deionized water) are prepared.

- **Optimization of Parameters:** The effect of solution pH (range 2–8) is investigated, with pH adjustments made using 0.1 M HCl or NaOH. Adsorbent dosage is varied from 0.5 to 5.0 g/L, and contact time is monitored from 10 to 180 minutes.
- **Equilibrium and Kinetics:** Flasks are placed in a thermostatic shaker incubator at 150 rpm. At predetermined time intervals, samples are withdrawn and filtered through 0.45 μm membrane filters. The residual concentration of Zn (II) in the supernatant is analysed using Atomic Absorption Spectroscopy (AAS).
- **Data Analysis:** The adsorption capacity (q_e, mg/g) is calculated using the mass balance equation:

$$q_e = \frac{(C_0 - C_e)V}{m}$$

where C_0 and C_e are the initial and equilibrium concentrations, V is the volume of the solution, and m is the mass of the adsorbent. These results provide the data necessary to model the adsorption kinetics and thermodynamics.

Adsorbent Preparation: From Agricultural Waste to High-Performance Media:

The systematic preparation of rice husk ash (RHA) is a critical determinant of its final adsorption capacity. The transformation process follows a strict protocol to ensure physical integrity, high surface area, and the desired surface chemistry necessary for the efficient sequestration of Zn (II) ions.

1. Pre-treatment and Thermal Processing:

Raw rice husks are initially subjected to a rigorous cleaning process to remove extraneous organic matter, soil particles, and field contaminants. The husks are washed multiple times with deionized water until the washings remain clear. The cleaned biomass is then oven-dried at 105°C for 24 hours to ensure complete moisture removal, which prevents uncontrolled steam-induced structural changes during subsequent thermal stages.

The dried rice husk is then transferred to a muffle furnace for controlled combustion. The temperature is ramped steadily to 600°C and maintained for a duration of 4 hours. This temperature range is crucial: it ensures the near-complete oxidation of organic cellulose and lignin components while preventing the sintering of silica, which would otherwise lead to a drastic reduction in the specific surface area and porosity. The resulting white or light-grey ash is sieved through a standardized mesh to obtain a uniform particle size distribution (e.g., $75 - 150\ \mu$m), ensuring consistency in batch experiments.

2. Chemical Modification Procedure:
While unmodified RHA exhibits inherent adsorption potential, its capacity is often limited by a sparse distribution of active functional groups. To improve this, a portion of the ash is subjected to chemical functionalization.

Adsorbent Activation: Enhancing Surface Reactivity:
The activation of rice husk ash (RHA) is a pivotal stage in developing a high-performance biosorbent. While raw RHA possesses a porous structure, its adsorption capacity for Zn (II) is often constrained by a lack of surface charge density and the presence of surface contaminants that block active pores. Chemical activation is specifically employed to overcome these limitations by restructuring the surface morphology and introducing functional groups that exhibit a high affinity for divalent metal ions.
The activation mechanism generally involves the treatment of the ash with either acidic or alkaline reagents. Alkali activation (typically using NaOH) is highly effective for silica-rich biomasses like RHA. During this process, the reagent promotes the partial dissolution of the silica matrix, which significantly increases the specific surface area and total pore volume. More importantly, this treatment facilitates the deprotonation of silanol (Si–OH) groups, resulting in a higher density of negatively charged sites ($Si–O^-$). These sites act as critical docking points for the cationic Zn (II) ions through electrostatic attraction and surface complexation. Furthermore, the activation process effectively removes mineral impurities and residual carbonaceous matter trapped within the pores during the combustion phase. By clearing these obstructions, the internal surface area becomes fully accessible to the adsorbate. Post-activation, the material is rigorously washed to neutralize the pH and remove residual chemical species that might otherwise interfere with the adsorption equilibrium. This transformation turns a passive agricultural byproduct into an engineered, high-affinity medium, drastically improving the kinetics and the ultimate adsorption capacity of the material in aqueous solutions.

Adsorption Kinetics: Modelling Zn (II) Uptake:
Adsorption kinetics are fundamental to understanding the mechanism of Zn (II) removal by rice husk ash (RHA), as they define the rate of uptake and the time required to achieve equilibrium. By analysing how the concentration of Zn (II) changes over time, we can determine the governing steps whether the process is controlled by film diffusion, intra-particle diffusion, or chemisorption. In this study, the kinetic data are typically analysed using Pseudo-first-order and Pseudo-second-order models. The Pseudo-first-order model assumes that the rate of occupation of adsorption sites is proportional to the number of unoccupied sites. However, for most biosorption processes involving metallic ions like Zn (II), the Pseudo-second-order model often provides a superior fit. This model suggests that the rate-limiting step is chemical adsorption (chemisorption), involving valence forces through the sharing or exchange of electrons between the RHA surface functional groups (such as silanol or hydroxyl groups) and the Zn (II) ions.
The linearized form of the Pseudo-second-order model is expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

where q_t and q_e represent the amount of Zn (II) adsorbed at time t and at equilibrium (mg/g), respectively, and k_2 is the rate constant.
A high correlation coefficient ($R^2 > 0.99$) for the Pseudo-second-order model indicates that the adsorption of Zn (II) onto both modified and unmodified RHA is dominated by chemical interaction. Furthermore, intra-particle diffusion plots are often used to determine if pore diffusion is a secondary rate-limiting factor. Understanding these kinetics is essential for scaling the process from laboratory batch experiments to industrial wastewater treatment applications, where contact time must be optimized to ensure high throughput efficiency.

Results and Discussion: Adsorption Performance and Mechanistic Insights:
The comparative performance of unmodified and chemically modified rice husk ash (RHA) for Zn (II) removal reveals significant improvements in both capacity and kinetics following chemical activation.
1. Impact of Modification on Adsorption Capacity:

Experimental data indicate that chemically modified RHA consistently outperforms unmodified RHA across varying initial metal concentrations. This enhancement is attributed to the alkaline treatment, which effectively increases the surface area and pore volume of the ash. Unmodified RHA typically demonstrates lower binding capacity due to surface impurities and a limited density of reactive sites. In contrast, the modified RHA exhibits a marked increase in the density of silanol (Si–OH) and hydroxyl functional groups. These groups provide a robust framework for binding Zn (II) ions through inner-sphere and outer-sphere complexation, confirming that surface functionalization is a critical step in optimizing the biosorbent's performance. Sivakumar, R., & Murugesan, V. (2005)., Tembhurkar, A. R., & Dongre, A. (2006).

2. Effect of pH and Optimization:

The solution pH is the most significant parameter influencing Zn (II) adsorption. Results demonstrate that at low pH, Zn (II) uptake is relatively poor; this is largely due to the competition between H^+ions and Zn^{2+}ions for the limited active sites on the RHA surface. Furthermore, at highly acidic pH levels, the adsorbent surface remains protonated, leading to electrostatic repulsion of the metal cations. As the pH increases toward the neutral range (pH 5.5–8.5), the surface becomes increasingly deprotonated, fostering a strong electrostatic attraction between the negatively charged Si–O⁻ sites and the divalent Zn (II) cations. Qiu, H., Lv, L., Pan, B. C., Zhang, Q. J., Zhang, P., & Zhang, Q. R. (2009).

3. Kinetic and Thermodynamic Behaviour:

Kinetic studies show that the adsorption process reaches equilibrium relatively rapidly, usually within 120 minutes. The high agreement with the Pseudo-second-order model ($R^2 > 0.995$) across both adsorbent types confirms that chemisorption is the rate-limiting step. The chemical modification not only increases the equilibrium adsorption capacity (q_e) but also enhances the initial rate of adsorption, likely due to better accessibility of active sites within the widened pore structures. Ofomaja, A. E. (2008)., Onyango, M. S., Kojima, Y., Matsuda, H., & Kenichi, O. (2004)., Pandey, P. K., Verma, Y., Kayal, S., & Sahu, S. (2009).

Thermodynamic analysis, calculated via the Van't Hoff equation, reveals that the adsorption of Zn (II) onto RHA is a spontaneous and endothermic process ($\Delta G^\circ < 0$ and $\Delta H^\circ > 0$). The increase in entropy ($\Delta S^\circ > 0$) suggests an increase in randomness at the solid-liquid interface during the adsorption of metal ions. Together, these results confirm that chemically modified RHA is an efficient, sustainable, and economically viable alternative for the remediation of zinc-contaminated aqueous solutions. Saha, B., & Orvig, C. (2010)., Saikia, N., & Parthasarathy, G. (2010).

To assist in the development of your research paper, I have identified key foundational and influential studies relevant to the adsorptive removal of heavy metals (specifically focusing on Zn (II) and similar divalent ions) using low-cost agricultural biosorbents like rice husk and its ash, published within the requested 2000–2019 period.

Fig. 1 : Effect of pH on adsorption of Zn(II) by Rice Husk Ash (unmodified) at different time intervals
Particle size < 55 µm; Temp.- 30±0.5°C; Conc. = 1.85×10–4m

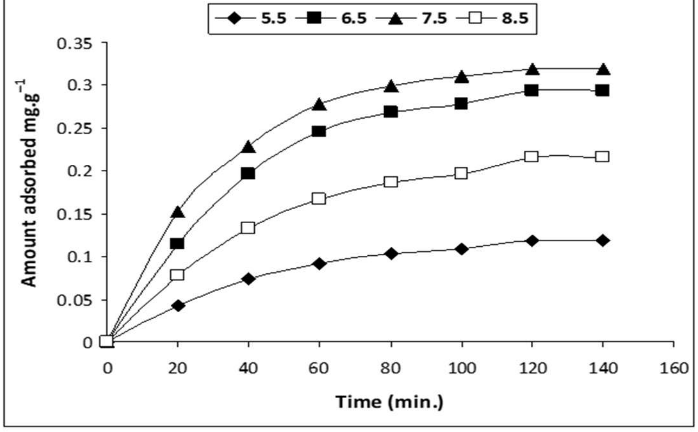


Fig. 2.: Effect of pH on adsorption of Zn(II) by Rice Husk Ash (modified) at different time intervals
Particle size < 55 µm; Temp.- 30±0.5°C; Conc. = 1.85×10–4m

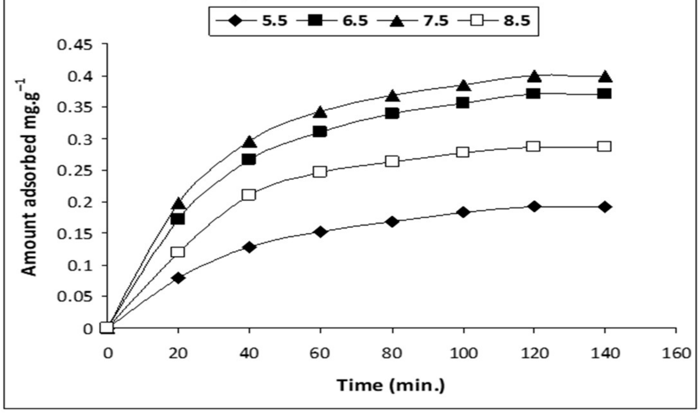


Fig. 3. : Rate constant plot for adsorption of Zn (II) on Rice husk ash (unmodified) at different pH.

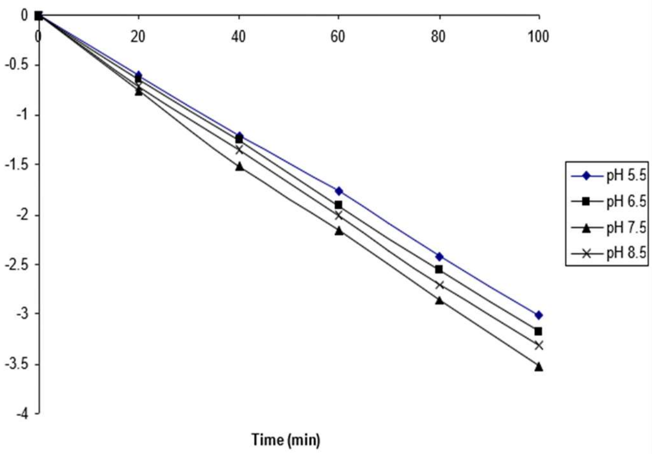
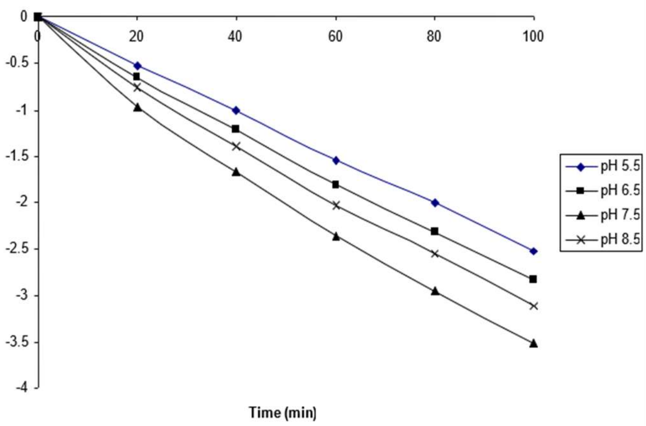


Fig. 4. : Rate constant plot for adsorption of Zn (II) on Rice husk ash (modified) at different pH.



Effect of pH on Metal Adsorption:

The adsorption of Zn (II) metal by using Rice Husk Ash (RHA) chemically unmodified and modified at pH range of 5.5 to 8.5 which are shown in Fig 1, Fig.2 , Fig.3 and Fig.4 is also explain the adsorption kinetics of Zn metal in reference to effect of pH. The pH of the adsorbent-adsorbate system which is a critical parameter which influencing the rate, extent and mechanism of adsorption. The pH values rise; the adsorption of metal ions also increases while that of anions decreases. The adsorption mechanism of H^+ and OH^- ions in the solution followed by acid-base dissociation. The adsorption of Zn (II) at different pH levels (5.5, 6.5, 7.5 and 8.5) using Rice Husk Ash at a constant temp. of 30±0.5oC concentration Conc. = $1.85\times10^{-}$ 4m Particle size < 55 mm; at pH 7.5, the percentage of adsorption, the unmodified rice husk ash achieved 60.95% at pH 7.5 while the modified rice husk ash as 77.65% (Fig.2). The research finding indicate that the adsorption process follows that first order reversible kinetics, the linear plots of (1-ut) versus t. The overall rate constant K^1 was determined from the slopes of these plots (Fig.3 & Fig.4). So, the findings indicate that the chemically modified adsorbents are increase removal rate in comparison to unmodified adsorbent due to specific surface area of the adsorbents.

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