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Glucose Derivative Functionalized Rice Husk Biochar for the Removal of Emerging Contaminant through Adsorption

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ABSTRACT

Carbazole (CBZ), recognized as a persistent contaminant with diverse ecotoxicological effects, presents a formidable challenge in its removal from wastewater. This study centers on the utilization of glucose derivative functionalized biochar (GDRB) as an adsorbent for the removal of CBZ. The synthesis of GDRB involved the cross-linking of β-cyclodextrin with rice husk biochar. The primary aim of this research was to evaluate the removal efficiency of CBZ from aqueous solutions using GDRB. In pursuit of this goal, a batch mode adsorption was implemented by employing synthetic wastewater spiked with CBZ. Various parameters, including the adsorbent loading, CBZ concentration, and treatment time, were systematically investigated for their influence on CBZ removal, with analysis performed using ultraviolet-visible spectroscopy. The results demonstrated that GDRB exhibited a superior ability to interact and adsorb CBZ compared to the original rice husk biochar. Specifically, GDRB showcased an impressive adsorption capacity of 17.45 mg/g, surpassing the original rice husk biochar (3.0 mg/g) and its activated form (7.0 mg/g) under the optimal GDRB loading of 0.04 g, initial CBZ concentration of 40 mg/L, and treatment time of 210 min. This setting yielded an outstanding CBZ removal efficiency of 87.25%. The findings provide valuable insights into the sustainable application of GDRB for treating emerging contaminants in wastewater.

1. Introduction

Emerging contaminants including medications, pesticides, industrial chemicals, detergents, and hygiene items are regularly encountered in water sources, food sources and dangerously accumulated in the effluent of wastewater treatment plant [1]. Carbazole (CBZ) is a heterocyclic aromatic compound and is widely present in the environment anthropogenically and exhibit both

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toxic properties and mutagenic, and it is categorized as "Benign tumorigen" [2]. The substance can enter the body in several ways, including ingestion, inhalation, and skin contact [3]. Various wastewater sources have been found to contain CBZ with concentrations ranging from 0.5 to 12.5 mg/L, including coking, biomass gasification, medications, sewerage, wood stabilizer wastewater, and urban landfill leachates [4]. Specifically, in coking effluent and wastewater from petroleum refineries, CBZ concentrations have been found between 7.8 and 12.5 mg/L [4]. Since the concentration of CBZ in wastewater is at a trace amount, necessitating a more potent adsorbent for efficient contaminant removal.

Emerging contaminants can be eliminated through the adsorption process, employing various strategies based on the characteristics of the pollutants. Adsorption technology utilizes cost-effective materials, demands low energy, offers flexibility, is user-friendly with adaptable designs and can function across various scenarios for water purification [5-7]. Various types of adsorbents can be used for removing the contaminants from wastewater including conventional ones such as activated carbon and non-conventional ones such as biosorbent and agricultural wastes [8]. A type of biosorbent derived from the glucose monomer, known as β-cyclodextrin, has received significant consideration due to its excellent hydrophobic cavity structure and numerous functional groups outside the macrocyclic rings that can be modified. These properties make it an attractive option for removing emerging contaminants from wastewater [9]. β-cyclodextrin could create distinct host-guest complexes by enclosing pollutants, but so far, cross-linked β-cyclodextrin complex have shown inferior adsorption capability than the traditional activated carbons due to their low surface areas [10]. Nonetheless, the high-water solubility of the β-cyclodextrin monomer has limited its usage in separating contaminants from aqueous solutions [9] rendering possibility for recovery after the treatment.

There are several works documenting β-cyclodextrin as an adsorbent [11,12]. Some of the work focused on the modification of this glucose derivative to improve its stability in water. β-cyclodextrin was reportedly incorporated with insoluble materials such as polymers, nanofibers, agricultural waste, and nanoparticles to adsorb heavy metal [13], nanometallic [14], phenol [15], and uranyl ions [16]. However, none of this adsorbent complex is tested on CBZ. The adsorption process parameter and performance of β-cyclodextrin complex with rice husk biochar has not been fully explored in this matter. The skins and cores of fruits, along with husks, and shells of beans and cereals derived from agricultural and agro-industrial wastes, offer significant benefits in the treatment of emerging contaminants. The presence of hemicellulose (15-30%), cellulose (35-50%) and lignin (20-30%) make these materials are easily available and chemically stable [17].

This study aims to investigate the efficacy of β-cyclodextrin, a glucose derivative, functionalized onto rice husk biochar (GDRB) for the treatment of CBZ from synthetic wastewater through batch adsorption. GDRB was synthesized by crosslinking β-cyclodextrin with acid-treated rice husk biochar, employing citric acid as a crosslinking agent. To assess its potential, the characteristics of GDRB were compared with those of raw rice husk biochar (RHB) and activated rice husk biochar (AcHB). The study systematically explores the influence of adsorbent dosage, initial CBZ concentration, and contact time on adsorption capacity of GDRB and CBZ removal efficiency.

2. Methodology

2.1 Materials

RHB was obtained from Tanegoodies Enterprise, while a range of chemicals, including phosphoric acid (99%), sodium hydroxide (≥98%), citric acid (99%), β-cyclodextrin (≥97%), potassium dihydrogen phosphate (≥99%), methanol (99.8%), and CBZ (≥95%), were supplied by Sigma-Aldrich.

2.2 Preparation and Characterization of GDRB
2.2.1 Activation of RHB

The RHB activation process was initiated by washing the RHB with deionized water to eliminate any impurities. Subsequently, the RHB underwent oven-drying at 60 ℃ for a duration of 24 hr. Following this, 2.5 g of the dried RHB was soaked in 25 mL of 50% phosphoric acid for a period of 12 hr. After the soaking phase, the RHB was filtered and thoroughly rinsed with 0.1 M sodium hydroxide solution and deionized water until the liquid pH became neutral, ensuring the removal of excess phosphoric acid. The resulting cleaned biochar was then dried at 60 ℃ until reaching a stable weight, ultimately yielding the AcHB.

2.2.2 Functionalization of AcHB

To functionalize the AcHB, a modification powder was prepared. β-cyclodextrin, citric acid and potassium dihydrogen phosphate were dissolved in 45 mL of deionized water at 3:3:1 ratio, respectively, with a total mass of 3.5 g. The resulting solution underwent heating at 140 ℃ in an oven for 5 hr, ensuring complete evaporation of the water. The dark brown solid product obtained was then fragmented into small particles through milling, followed by multiple rinses with deionized water. Subsequently, the milled solid was oven-dried at 60 ℃ for 12 hr to yield modification powder.

The preparation of GDRB was followed the procedure outlined by Azman *et al.,* [18] and Jiang *et al.,* [19] with some adjustment. A mixture was created by combining 250 mL of deionized water containing 2.5 g of modification powder with 1.25 g of AcHB. This mixture underwent ultrasonication at a temperature of 60 ℃ for 5 min. Subsequently, the biochar was filtered and washed with deionized water to eliminate any excess modification powder. The GDRB was produced by drying the biochar at 60 ℃ until its weight reached a stable state.

2.2.3 Characterization of rice husk-based adsorbent

For characterization, the surface configuration of rice husk-based adsorbents (RHB, AcHB, and GDRB) were observed using scanning electron microscopy (SEM, Hitachi, TM3030). Prior observation, sample were dried for 12 hr at 60 ℃ and gold-coated under a vacuum environment to enhance the images' quality.

2.3 Synthetic Wastewater Preparation

Initially, a predetermined amount of CBZ was dissolved in pure methanol to create a standard stock solution with a concentration of 100 mg/L. To prevent photodegradation, the standard solution was tightly capped and stored in a dark area. The desired working concentrations were achieved by diluting the stock solution while keeping a constant methanol concentration of 30% [20].

2.4 Batch Adsorption

Batch mode adsorption experiments were conducted in 50 mL conical flask to explore the impact of various operating parameters on the adsorption capacity of GDRB and the percentage removal of CBZ. The parameters encompassed GDRB dosage, initial CBZ concentration and contact time, with Table 1 illustrating the upper and lower levels assigned to each factor. These values were chosen based on insight gained from previous experiments (unpublished work).

Before delving into the influence of each operating parameter on CBZ adsorption, an assessment of the adsorption potential of different rice husk-based adsorbents (RHB, AcHB and GDRB) was carried out. This preliminary experiment involved an adsorbent dosage of 0.02 g, an initial CBZ concentration of 30 mg/L and a contact time of 120 min.

Table 1
Operating parameters and levels used for CBZ adsorption using GDRB

Run	Operating parameter	Level		Fixed parameter
		Low	High	
1-5	GDRB dosage (g)	0.02	0.1	i. Initial CBZ concentration (mg/L): 30. ii. Contact time (min): 120.
6-10	Initial CBZ concentration (mg/L)	10	50	i. GDRB dosage (g): Optimal level from run 1-5. ii. Contact time (min): 120.
11-20	Contact time (min)	30	300	i. GDRB dosage (g): Optimal level from run 1-5. ii. Initial CBZ concentration (mg/L): Optimal level from run 1-5.

The experimental procedure involved introducing a predetermined amount of rice husk-based adsorbent and 20 mL of a known CBZ concentration solution at pH 7 into the conical flask. The mixture was subjected to agitation at 250 rpm and a temperature of 25 °C using an orbital shaker. After a specified duration, the solution underwent filtration through syringe filter and the equilibrium CBZ concentration was determined using an ultraviolet-visible spectroscopy at a wavelength of 293 nm. The adsorption capacity of rice husk based-adsorbent (q_e) and the CBZ removal efficiency were subsequently calculated using the following equations:

$$q_e\left(\frac{mg}{g}\right) = \frac{(C_0 - C_e)V}{m} \quad (1)$$

$$R(\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

where C_0 and C_e (mg/L) are the initial and equilibrium concentrations of CBZ, respectively. V (L) represents the volume of the CBZ solution and m (g) is the rice husk-based adsorbent dosage [21].

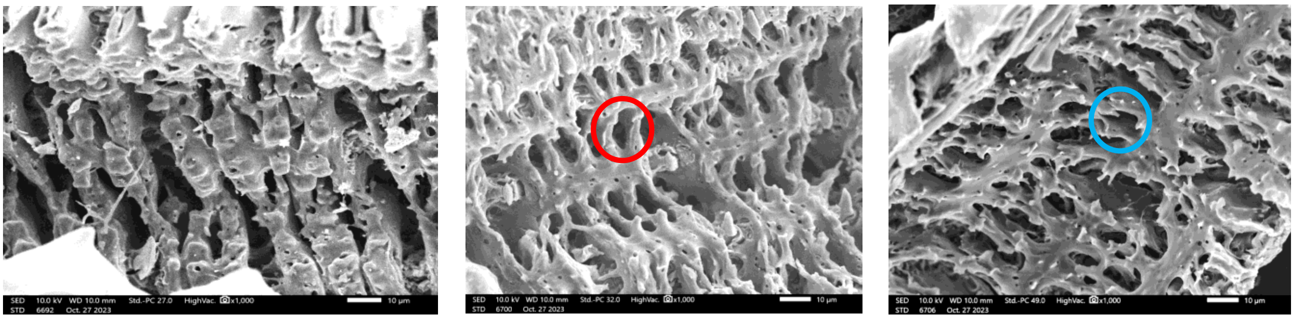
3. Results

3.1 Adsorbent Characterization

The SEM images depicting various types of rice husk based-adsorbent are illustrated in Figure 1. From the figure, RHB exhibits smooth lateral surfaces and long channels with limited pores. The formation of pores was due to the pyrolysis of rice husk which had contributed to a sudden rise in surface area of the biochar [22]. Through the heating process, pores are materialized on the biochar's surface, accompanied by the generation of volatile substances. These transformations collectively contribute to the modification of the biochar's physical characteristics. With the presence of these pores, RHB can serve as an adsorption medium [23].

A comparison was made between RHB and AcHB, revealing that the latter exhibited a surface characterized by greater roughness and porosity, as illustrated in Figure 1(b). Phosphoric acid, known for its dehydrating properties, plays a crucial role in removing generated tar and other volatile products during pyrolysis of RHB. The phosphoric acid undergoes dehydration during activation and persists as salts of phosphoric acid deep within the porous structure of the RHB, occupying significant volumes. After thorough washing following preparation, a significant level of microporosity is created

[24]. Besides, treating the biochar with organic acid enhanced its porosity by separating loosely bonded components, such as labile carbon and volatile matter, from its surface [18].



(a) (b) (c)

Fig. 1. SEM images (a) RHB (b) AcHB (c) GDRB. The red circle in the images highlights the formation of new pores in AcHB, while blue circle indicates the formation of glucose derivatives in GDRB

Figure 1(c) shows the cross-sectional images capturing the structural features of GDRB. SEM observations disclosed the presence of varied-sized pores on the surface of GDRB which indicates the complex pore formation process. Moreover, the resulting GDRB has a smoother surface than AcHB. This observation might be due to the incorporation of the glucose derivative into the grooves on the surface of AcHB during the functionalization [25]. These alterations decreased the biochar's surface area and porosity. The result was in line with the findings reported by Rajandran and Nasratun [26], but the latter used epichlorohydrin as a crosslinker.

3.2 Adsorption Performance of Different Rice Husk-Based Adsorbents

The removal of CBZ from a 30% methanol solution was assessed using three different types of adsorbents: RHB, AcHB, and GDRB. As shown in Figure 2, GDRB exhibited the highest adsorption capacity and removal efficiency among the three adsorbents, at 8.34 mg/g and 28.57%, respectively. In contrast, RHB showed the lowest adsorption capacity and removal efficiency, with values of 3.03 mg/g and 10.99% whereas the adsorption capacity (6.75 mg/g) and removal efficiency (23.24%) of AcHB are higher than RHB.

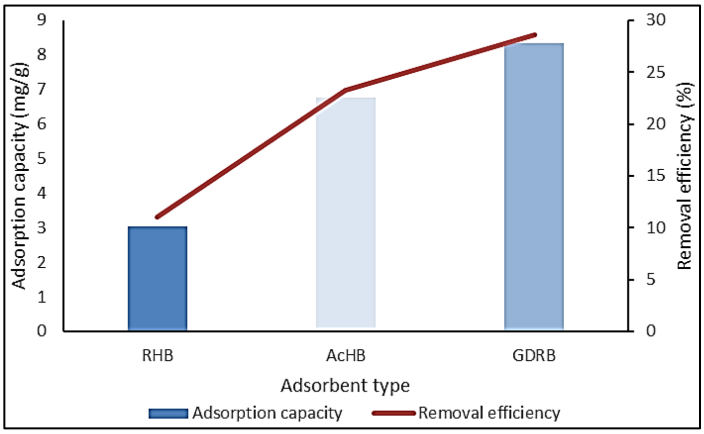


Fig. 2. Effect of different types of adsorbents

AcHB exhibits better performance than RHB, as the pore quantities increase with the addition of new pores through chemical activation using phosphoric acid. The adsorptive capacity and bulk density of activated carbons derived from lignocellulosic materials are also optimized [27]. The combination of impregnation with phosphoric acid and subsequent washing steps is described by Azman *et al.,* [18] as a process that effectively removes impurities and opens closed pores. This process contributes to an increased surface area, which is often desirable for applications where a high surface area is crucial, such as in adsorption processes.

GDRB shows the best performance among the three adsorbents as the inherent glucose derivatives (β-cyclodextrin) possess a varying number of hydroxyl groups capable of engaging in chemical reactions to bind a diverse range of contaminants. The added substituents can beneficially enhance the inclusion of contaminants through host-guest complexation and, in comparison to native cyclodextrins, improve the capacity to solubilize and stabilize these molecules [28]. CBZ is an aromatic compound, containing benzene ring that can readily slip into the torus of cyclodextrin. This is because the interior part of this glucose derivative is hydrophobic while the external part of the torus is hydrophilic creating a complementary relationship. Besides, interactions occur with the functional groups formed as a result of functionalization [25].

This finding aligns with the results from Azman *et al.,* [18], where the CBZ removal rate by the starch derivative-functionalized rice husk biochar was 98.01%, followed by acid-treated rice husk biochar at 23.3%, and lastly, raw rice husk biochar at 23.0%. The authors also deduced that the heightened adsorption capacity of starch derivatives complex adsorbent is attributed to the numerous hydroxyl groups and the inner core of β-cyclodextrin within the hydrophobic area, allowing for the formation of complexes with CBZ, an aromatic compound.

3.3 Effect of GDRB Dosage

Figure 3 illustrates the CBZ removal corresponding to GDRB dosage. Both adsorption capacity and removal efficiency were linearly improved as the GDRB dosage increased from 0.02 to 0.04 g, at a maximum of 12.17 mg/g and 80.85%, respectively. Beyond this point, the adsorption capacity decreased to the lowest of 4.98 mg/g and the removal efficiency stabilized at an average of 76%.

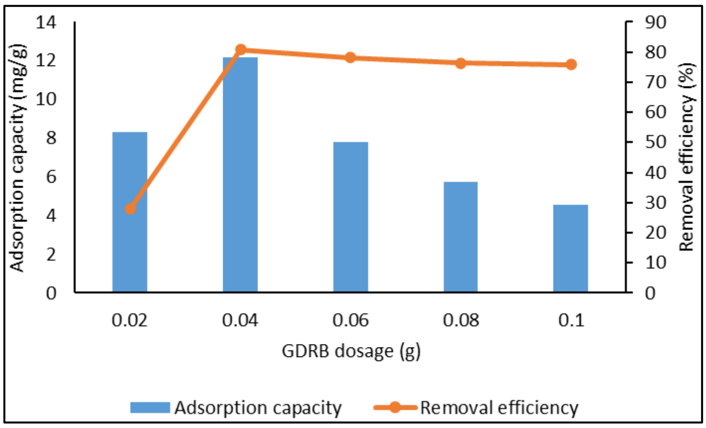


Fig. 3. Effect of GDRB dosage

Improvement in the removal efficiency is attributed not only to the increased number of active sites available for adsorption, which facilitates the adsorption of CBZ but also to an expansion in the adsorbent surface area [29]. However, with a subsequent increase in adsorbent dosage, the removal efficiency becomes stable. This stability can be attributed to the limiting number of contaminants that can attach to the rich active sites of GDRB, creating an excess free active site [21]. On the other hand, the declining trend in the adsorption capacity is due to the overlapping and aggregation of GDRB active sites, leading to a decrease in the number of CBZ molecules per unit adsorbent. These findings align with the study done by Zafar *et al.,* [30], investigating the adsorption capabilities of rice husk biochar on copper (II) ions. From the figure, the maximum removal efficiency was reached at 0.04 g of GDRB, hence, the subsequent experiment will fix the adsorbent dosage to this amount.

3.4 Effect of Initial CBZ Concentration

Figure 4 displays how different initial CBZ concentrations affect the adsorption performance. A notable trend in the adsorption capacity and removal efficiency of CBZ can be observed. As the initial concentration of CBZ was systematically elevated from 10 to 50 mg/L, the adsorption capacity exhibited a substantial increase, rising from 1.86 mg/g to 16.96 mg/g. This suggests a positive correlation between the initial concentration of CBZ and its adsorption capacity. However, the removal efficiency has a slight difference in the trend where it initially increased from 37.19% to 81.83% as the initial concentration of CBZ increased from 10 mg/L to 40 mg/L. The removal efficiency then experienced a decline to 67.08% at 50 mg/L.

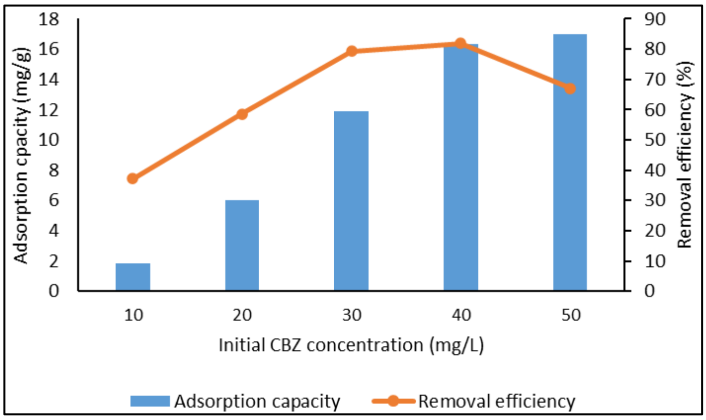


Fig. 4. Effect of initial CBZ concentration

As the initial concentration of CBZ increased, the driving force for surface diffusion between CBZ and GDRB intensified, thereby contributing to a higher adsorption capacity. However, the adsorption capacity slowly reached a plateau as CBZ concentration approached 50 mg/L, indicating that the saturation of adsorption sites on the surface of GDRB, preventing the accommodation of a higher concentration of CBZ [31]. On the other hand, the removal efficiency decreases after reaching the equilibrium point (40 mg/L of CBZ). This decrease is attributed to the abundance of CBZ molecules, which led to limitations on the adsorption process. This phenomenon arises from the competitive adsorption between CBZ molecules for the available sites, as the existing active sites for adsorption on the materials are constant.

Consequently, this competition causes a decrease in the overall removal efficiency [32,33]. A similar pattern emerges when Chang *et al.,* [34] investigated cyclodextrin-based polymer networks targeting organic dyes. From their study, the adsorption capacity showed a noticeable increase within the dye concentration range from 0.01 to 0.05 mmol/L of methylene blue. However, as it reached 0.05 mmol/L the adsorption capacity was slowed down and eventually levelled off. This similarity in the observed trends between Chang *et al.,* [34] work and the present study suggest a common behavior in the adsorption, highlighting the relevance and consistency of these findings across different research efforts.

3.5 Effect of Contact Time

This study explores the impact of contact time on the removal of 40 mg/L of CBZ using 0.04 g of GDRB dosage. Figure 5 shows the removal efficiency and adsorption capacity over 300 min contact time. Approximately 72% of total CBZ was rapidly removed within the first 150 min, at a maximum rate of 0.19 mg/min. Subsequently, the adsorption capacity increased steadily as contact time reached a plateau after 210 min, and then remaining constant. Hence, the equilibration time was established as 210 min, deemed sufficient for CBZ removal by the adsorbent with an adsorption capacity of 17.45 mg/g and a removal efficiency of 87.25%. The profile in Figure 5 exhibits a smooth and continuous pattern, indicating saturation and suggesting the potential monolayer coverage of CBZ on the adsorbent surface. The increase in removal efficiency and adsorption capacity is attributed to the abundance of available binding sites on GDRB, with mesopores nearly saturated during the initial stage of the adsorption process. As the process continues, CBZ must traverse farther and deeper into GDRB micropores, encountering greater resistance, leading to a decrease in driving force and adsorption rate [32].

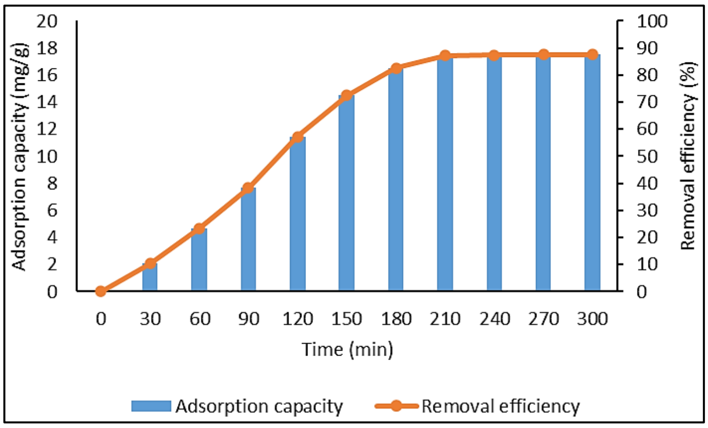


Fig. 5. Effect of contact time

A parallel trend is evident in Chang *et al.,* [34] investigation focusing on cyclodextrin-based polymer networks and their interaction with organic dyes. Their findings concerning the adsorption rate of organic dyes closely mirror those of this study, with both studies achieving a substantial portion, over 50%, of the total adsorption within the initial 2 hr. This initial high adsorption rate gradually declined over time in both cases. However, there is a notable distinction in the time required for each study to reach the equilibrium stage. The study conducted by Chang *et al.,* [34] took a longer duration, 8 hr, in contrast to the 3.5 hr of the current study. This discrepancy in equilibration times may be attributed to variations in the targeted pollutant and the initial concentration of the pollutant, highlighting the influence of these factors on the adsorption kinetics.

4. Conclusions

CBZ was successfully adsorbed onto GDRB. Prior to investigating the impact of operating parameters on CBZ adsorption, the adsorption performance of this glucose derivative complex was compared with other rice husk-based adsorbents, namely RHB and AcHB. This comparison aimed to show that each modification on RHB results in a better adsorption capacity and removal efficiency. Out of the three adsorbents, GDRB shows the best adsorption capacity, and removal efficiency, followed by AcHB and RHB. The best condition that maximized the adsorption performance of GDRB within the studied range was 0.04 g of adsorbent dosage to treat 40 mg/L of CBZ for 210 min. The highest adsorption capacity and removal efficiency obtained under these conditions were 17.45 mg/g and 87.25%, respectively. In the future, an emphasis should be placed on the examination of kinetics, thermodynamics, and isotherms of GDRB concerning CBZ adsorption, as well as other heterocyclic aromatic hydrocarbons.

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