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Nutrient Capture and Circular Solutions: Zinc Chloride Activated Bamboo Biochar for Aquaculture Wastewater Nitrate Recovery

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The growth of aquaculture farming has produced more nutrient-rich wastewater. This wastewater contributes to eutrophication in the water bodies. Meanwhile, the bamboo furniture industry generates large amounts of solid waste biomass wastes. Bamboo biomass waste could be used in many valuable ways because it is abundant, and has high strength and cellulose content, but it is still mostly underused and often ends up in landfills. To reduce water pollution and nutrient buildup caused by aquaculture wastewater (AW), capturing nutrients with bamboo-derived biomass offers a sustainable circular solution that turns waste into a valuable resource. This study explores the efficiency of zinc chloride ($ZnCl_2$) activated bamboo biochar (ABB) to recover nitrate from AW. ABB was prepared using different impregnated ratios of $ZnCl_2$ at 500 °C. The best prepared condition was further optimized for adsorption process by varying the dosage of ABB and contact time. Results shows that the most efficient nitrate adsorption was 68.8 % when $ZnCl_2$ ABB was prepared at 500 °C using an impregnation ratio of 3. At optimum process parameter (2.5 g, 150 rpm and 90 min) the adsorption efficiency and capacity were further enhanced to 82.8% and 0.676 mg/g respectively. The adsorption process was well represented by Freundlich and pseudo second-order model. The adsorption efficiency decreased to 63.2 % when tested with actual AW due to other competing contaminants in the actual wastewater. This study demonstrates the potential of ABB as a sustainable nitrate recovery material from AW. Spent ABB could be further exploited as soil conditioner. This study contributes to circular economy practices by promoting nutrient recovery from wastewater and the reutilization of waste biomasses.

1. Introduction

Aquaculture has transformed into the primary aquatic animal producer as it has overtaken conventional fishery output in 2022 (FAO, 2024). Unfortunately, conventional aquaculture operations frequently result in secondary contamination and a degradation of water quality, which poses a threat to available aquatic resources. Untreated aquaculture wastewater (AW) containing high nitrate level can cause severe water pollution, including high nutrient loads and destructive algae blooms known as eutrophication (Tom et al., 2021). This dissolved nitrate if recovered can be reutilized as soil conditioners. Meanwhile, Malaysia has abundant bamboo resources, with an estimated total of 33 million tonnes (Patrick, Ward & Kashio, 1994). Although Malaysia has plenty of bamboo, its industry is still underdeveloped and largely produces low-value products (Chaudhary et al., 2024). Moreover, most of the waste bamboo biomass from the bamboo manufacturing industry is left unutilized and landfilled (Li and He, 2019). Thus, there is a need to reutilize bamboo biomass waste and recover nitrate from AW.
Adsorption is a potential method to remove or recover pollutants from water bodies. Biochar has been reported as prominent adsorbent for nitrate in water. For example, biochars derived from banana peel (Reddy et al., 2015), orange peel (Dey et al., 2022), hazelnut shell (Stjepanović et al., 2022) and sugarcane bagasse (Divband Hafshejani et al., 2016) biochars has been successfully modified to remove nitrate from synthetic solution of nitrate. Another study by Viglašová et al., 2018, has shown that bamboo biochar modified with montmorillonite could remove 5 mg/g of nitrate from synthetic wastewater. So far activated bamboo biochar (ABB) is yet to be

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tested for nitrate recovery. Thus, this study aims to evaluate the ability of Zinc Chloride ($ZnCl_2$) activated bamboo biochar for recovery of nitrate from synthetic and actual AW.

2. Methods

2.1 Aquaculture wastewater sampling and synthetic wastewater preparation

Actual AW was collected from a fishing pond in Malim Nawar, Perak, Malaysia (geographical coordinate: 4.3614, 101.15585). This site was selected because the wastewater is discharged into the nearby river. These samples were clearly labelled and refrigerated at 4 °C to preserve their characteristics for accurate results. To ensure consistency, only one batch of water sample was collected and used for the entire experiment. Before each experiment, samples were drawn and allowed to reach room temperature for minimum 3 hours. The initial nitrate concentration in the AW was measured using UV-vis Spectroscopy DR3900 Hach. The initial nitrate concentration in the AW upon collection was recorded as 3.8 mg/L (± 0.5 mg/L) on average. Synthetic nitrate infused wastewater was prepared using potassium nitrate (KNO_3) powder (Bendosen, N-Total: 13.4 %). A concentration of 100 mg/L of nitrate solution was prepared by dissolving 0.10 g of KNO_3 in 1 L of distilled water. This solution was further diluted to a concentration of 10 mg/L for the adsorption test. All experiments were conducted in triplicate.

2.2 Activated bamboo biochar preparation

Bamboo biochar was obtained from SEAD Industries Sdn. Bhd. The bamboo biochar was prepared at 800 °C in limited oxygen. The biochar was sieved to a size of 0.106 mm to 0.125 mm. The sieved biochar (15 g) was added to various weight (%) ratios (1:1, 1:2 and 1:3) of $ZnCl_2$ (PC lab reagent, purity: 98.5 %). The samples are labelled as ABB1, ABB2 and ABB3. The impregnation was done using an orbital shaker for 3 h at a mixing speed of 180 rpm. Next, the impregnated biochar samples were dried in an oven at 105°C for 12 h (Sahoo et al., 2021). Upon drying, the biochar samples were pyrolyzed at 500°C for 1 h at a heating rate of 10°C/min under nitrogen environment. After modification, thorough washing with deionized water was conducted to remove residual $ZnCl_2$ and its by-products until the pH of the filtrate was neutral. Then it is dried in the oven at 105°C for 12 hr. Upon cooling, the samples are store in air-tight container prior to usage. The wash water was neutralized to pH 6.5–8.0 before disposal, ensuring compliance with environmental discharge standards. Alkaline neutralization method was used to recover zinc hydroxide, preventing direct discharge.

2.3 Adsorption mechanism, kinetics and characterization

The surface functional groups of ABB were analyzed using the Attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy (ATR) -FTIR (Perkin-Elmer Spectrum 2) technique. Adsorption studies were started with synthetic wastewater followed by actual AW after optimizing the operating conditions. Initial run was done to identify the best impregnation ratio of $ZnCl_2$, followed by optimizing the ABB dosage (0.5 - 2.5 g) and contact time (30 - 300 min). For the effect of dosage, the mixing speed (150 rpm) and contact time (90 min) was fixed. For the effect of contact time, the dosage and mixing speed was fixed as 2.5 g and 150 rpm. The adsorption process was carried out using 200 mL of a 10 mg/L nitrate solution, prepared in a conical flask and agitated on an orbital shaker. Each testing was repeated twice and average readings were reported. The adsorption efficiency and capacity were calculated using Eq(1) and Eq(2) (Battas et al., 2019).

$$\textit{Adsorption efficiency (\%)} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

$$\textit{Adsorption capacity, q(mg/g)} = \left(\frac{C_i - C_f}{m}\right) \times V \quad (2)$$

To describe the adsorption mechanisms, Langmuir and Freundlich isotherm models were used. The linearized form of the Langmuir and Freundlich isotherm is shown in Eq(3) and Eq(4). To understand the kinetics of the adsorption process, pseudo first order (PFO) and pseudo second order (PSO) kinetic models were applied. The linearized form of the PFO and PSO equations are shown in Eq(5) and Eq(6) (Elemile et al., 2022).

$$\frac{1}{q_e} = \frac{1}{q_{max} \cdot K_L \cdot C_e} + \frac{1}{q_{max}} \quad (3)$$

$$Inq_e = InK_f + \frac{1}{n} InC_e \quad (4)$$

$$log(q_e - q_t) = log(q_e) - \frac{k_1}{2.303} t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{t}{q_e} \quad (4)$$

3. Results and discussion

3.1 Adsorption of Nitrate at Different Impregnation Ratio

Figure 1 shows the adsorption efficiency and capacity of nitrate ABB at various impregnation ratios (ABB1= 1:1, ABB2=1:2 and ABB3=1:3). The highest adsorption efficiency (68.8 %) and capacity (0.953 mg/g) was obtained by ABB3. Higher impregnation ratios increase the amount of $ZnCl_2$ on the bamboo biochar surface, promoting stronger activation and better pore development. $ZnCl_2$ activation can also enhance the oxygen-containing functional groups on the biochar surface Trivedi et al. 2025. It helps the adsorption process through chemical interactions. This was proven by FTIR results shown in Figure 2. A distinct peak around 3403 cm^{-1} was detected for bamboo biochar, which corresponds to the O-H stretching (hydroxyl); a peak near 1734 cm^{-1} is associated with a carbonyl group (C=O); and a smaller peak around 1037 cm^{-1} corresponds to the C-O stretching. These functional groups originate from lignocellulosic components (hemicellulose and lignin) that consist in the bamboo biochar (Chen et al., 2022). The peaks in ABB3 were similar to those of bamboo biochar, but showed clear differences in intensity and position. For example, at the peak around 2098 cm^{-1}, the alteration in the functional groups of ABB3 was due to the activation process which led to the shifting of the peak (Phothong et al., 2021). Furthermore, a peak about 1581 cm^{-1} indicates the consistency of vibration with aromatic C=C stretching. It has been noted that the presence of aromatic C=C stretch, aromatic C-H bend, and O-H stretch could also improve the nitrate adsorption onto the surface of ABB3 via electrostatic attraction (Eissa et al., 2024). Thus, for the subsequent process study ABB3 was used.

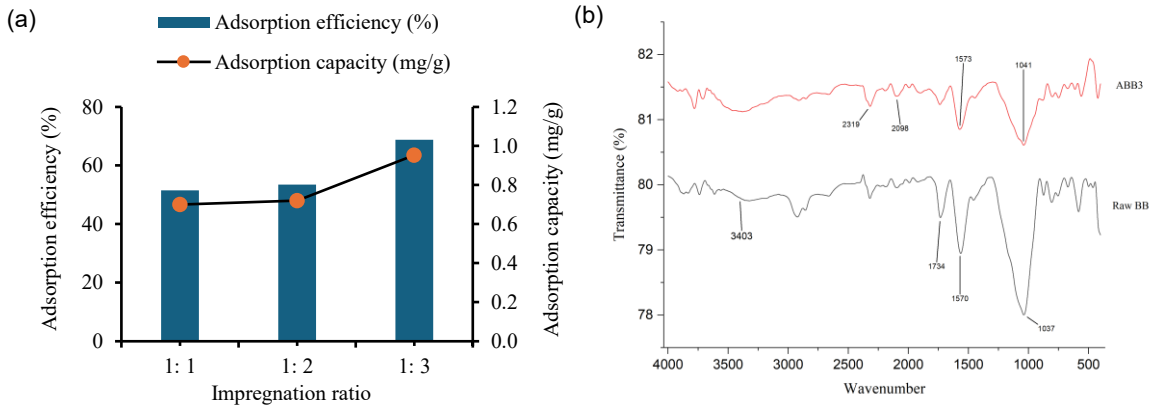

Figure 1: (a) Nitrate recovery efficiency and capacity by ABB at different impregnation ratio and (b) FTIR results of ABB3 and raw bamboo biochar

3.2 Effect of adsorbent dosage and contact time

Figure 3 illustrates the effect of different ABB3 dosage on the adsorption efficiency of nitrate in synthetic wastewater. A positive correlation between the dosage of ABB3 and the adsorption efficiency was observed, whereby as the dosage of ABB increased, the nitrate recovery was more efficient. For example, as the dosage of increases from 0.5 g to 2.5 g, the adsorption efficiency also increased from 58.3 % to 74.3 %. At lower dosage, the availability of active sites on the surface of activated bamboo biochar is limited, which hinders the capture and recovery of nitrate ions from the nitrate solution. However, at the higher dosage, nitrate recovery via adsorption from synthetic wastewater improved noticeably due to greater surface area and number of binding sites availability on ABB3 (Trivedi et al., 2025). The increment in adsorption efficiency from 0.5 g to 1.0 g is approximately 7 %, while the increment from 2.0 g to 2.5 g is about 3 %. This condition, referred to as diminishing returns, indicates that the nitrate concentration in the sample solution would become the limiting factor when the ABB3 is saturated (El-Shafey et al., 2024). Therefore, excessive adsorbent dosage has minimal effects on the adsorption activity due to the accumulation and saturation of active sites (Tyagi et al., 2022). As a result, adsorbent dosage of 2.5 g shows the best adsorption efficiency, but dosage of 0.5 g shows the best adsorption capacity (2.4 mg/g) among the others. Thus, a dosage of 0.5 g was used for contact time optimization.

Figure 4 shows the effect of contact time on nitrate adsorption efficiency. Nitrate recovery increases with contact time at first, then levels off after reaching a peak. At shorter contact time (30 min), the adsorption efficiency was relatively low at 59.3 %. This is due to inadequate contact time for nitrate ions to attach to the active sites available on the ABB3. Majority of active sites still remain unoccupied due to an incomplete adsorption process (Astuti et al., 2023). Incomplete adsorption process could limit the diffusion rate of nitrate ions into the internal pore structure of the ABB (Sutar and Jadhav, 2025). Nevertheless, when the contact time increased to 60 min,

the adsorption efficiency increased to 66.3 %. As expected longer contact time allows more nitrate ions to bind to the available sites, improving the interaction between the adsorbate and adsorbent (Erasga et al., 2024). At the contact time of 90 min, the adsorption efficiency had reached its peak at the value of 82.8 %. Further increase in time resulted in a negligible effect on the efficiency of nitrate adsorption. This is primarily caused by the saturation of ABB3 once equilibrium has been achieved. Therefore, a contact time of 90 min is sufficient to achieve the optimal adsorption capacity of 0.676 mg/g.

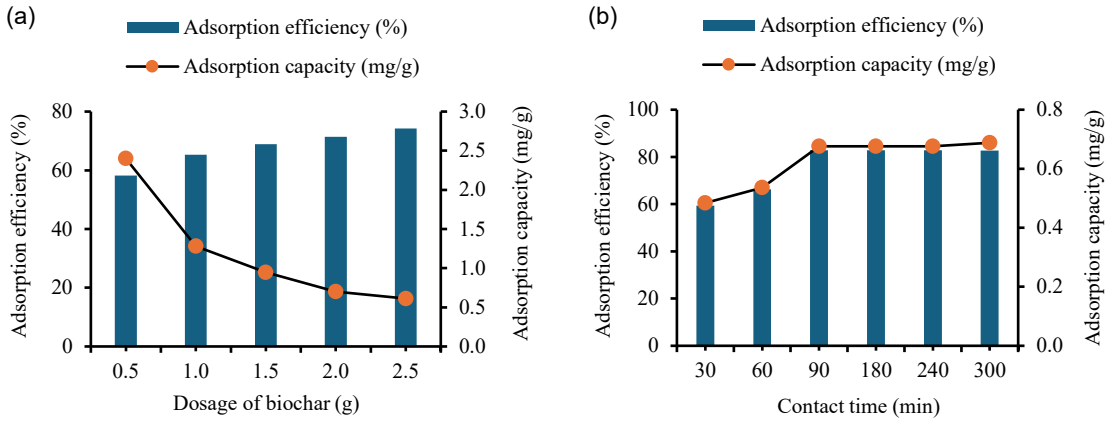

Figure 3: Nitrate recovery efficiency and capacity by ABB3 at various (a) dosage and (b) contact time

3.3 Adsorption mechanism and kinetics

The Langmuir and Freundlich isotherm models were analyzed to characterize the adsorption behavior. The results of the isotherm and kinetics parameters are shown in Table 1. The coefficient of determination (R^2) for both the Langmuir and Freundlich model shows > 0.9500 suggests a good linear fit to the experimental data. The Freundlich model provided a better fit compared to the Langmuir model. The adsorption intensity (n) of 0.3495 (<1) has been calculated from the regression equation for the Freundlich model, indicative of favorable and heterogeneous multilayer adsorption.

Table 1: Adsorption Isotherm and kinetics parameters

Isotherm	Parameter	Value	Kinetic Model	Parameter	Value
Langmuir isotherm	R^2	0.9559	PFO	R^2	0.6251
	q_m (mg/g)	-0.6466		q_e (mg/g)	0.4907
	K_L (L/mg)	-5.3595		K_1 (min^{-1})	0.0092
Freundlich isotherm	R^2	0.9922	PSO	R^2	0.9962
	n	0.3495		q_e (mg/g)	0.7235
	K_f	0.0368		K_2 ($g/mg \cdot min$)	0.0963

Parameter values obtained for Langmuir model were negative. As reported by Igwe and Abia (2007), the negative values formed might be due to experimental errors or a higher initial concentration of adsorbate used. Even after repeats similar results were obtained. The result suggests that the Langmuir model does not fit the experimental data well. Meanwhile, to analyze the kinetics of nitrate adsorption, two commonly used models *i.e.,* the PFO and PSO were employed. The kinetic parameter results are shown in Table1. The R^2 value of PSO was greater than PFO. This indicates that the PSO model correlated well with the experimental data. Although the equilibrium adsorption capacity (q_e) predicted by the PSO model (0.7235 mg/g) was higher than the experimental value (0.676 mg/g), it was much closer than the value predicted by the PFO model (0.4907 mg/g). The good fit to PSO model suggests that the adsorption process is likely governed by chemisorption. It is expected that the reaction of nitrate adsorption onto activated bamboo biochar with $ZnCl_2$ activation is expressed as:

$$Zn^{2+} + {NO_3}^- \rightarrow Zn^{2+} \ldots {NO_3}^-$$

Surface protonation of ABB3 by $ZnCl_2$ enhances the positive charge of the adsorbent, resulting in greater electrostatic attraction toward negatively charged nitrate ions (Liu et al., 2018; Sefatlhi et al., 2024). Moreover, active surface functional groups on ABB3 such as carboxyl, hydroxyl, and amine groups further facilitate bond formation with electronegative atoms, and enhance its nitrate adsorption capacity.

3.4 Effect of on actual aquaculture wastewater

The optimized ABB3 adsorbent from the synthetic wastewater trial was used to test the recovery activity of nitrate from AW. The adsorption process was examined under the same optimum parameters (dosage: 2.5 g; mixing speed: 50 rpm, and contact time: 90 min). The initial concentration of the AW was detected as 3.80 mg/L. The nitrate recovery efficiency and adsorption capacity were 63.2 % and 0.192 mg/g respectively, which were lower than those achieved with synthetic wastewater. According to Priya et al. (2022), actual AW contains various other pollutants, such as ammonia, phosphorus, and etc. These competing contaminants may reduce the number of available adsorption sites for nitrate on the surface of ABB3. Nevertheless, the results demonstrate that ABB3 is still capable of removing nitrate from actual AW.

4. Conclusions

Nitrate, a waste nutrient from AW, was successfully recovered using waste bamboo biochar activated with $ZnCl_2$ at a 1:3 ratio and a pyrolysis temperature of 500 °C. The highest recovery efficiency and adsorption capacity of 82.8 % and 0.676 mg/g, were achieved under optimal conditions of a 90 min contact time and a dosage of 2.5 g using synthetic wastewater. It was concluded that nitrate recovery through adsorption was facilitated by heterogeneous multilayer adsorption via chemisorption and surface functional groups. However, when applying the same optimized conditions to actual AW, the recovery performance was reduced to 63.2 % and 0.192 mg/g. This is due to competition from other coexisting pollutants. The recovery capacity was lower than values reported in the literature, likely due to the low initial nitrate concentration. It is generally more challenging to adsorb low-concentration pollutants from water, particularly in actual AW. Future studies should focus on identifying the role of co-existing pollutants in the nitrate recovery process by ABB. Spent or nitrate-recovered ABB has good potential as a soil conditioner, as $ZnCl_2$ supplies essential nutrients for plants and the biochar improves soil fertility and structure.

Nomenclature

AW – Aquaculture Wastewater
ABB – Activated Bamboo Biochar
ABB1 – Activated Bamboo Biochar (Ratio 1:1)
ABB2 – Activated Bamboo Biochar (Ratio 1:2)
ABB3 – Activated Bamboo Biochar (Ratio 1:3)
$ZnCl_2$ – Zinc Chloride
$\boldsymbol{C_i}$ – initial concentration of nitrate (mg/L)
$\boldsymbol{C_f}$ – final concentration of nitrate (mg/L)
m – mass of ABB (g)
V – volume (L)
q_e – adsorption capacity at equilibrium (mg/g),
q_{max} – maximum adsorption capacity (mg/g)
K_L – Langmuir constant related to the affinity of binding sites (L/mg)
C_e – equilibrium concentration of the adsorbate in solution (ppm)
K_f – Freundlich constant
n – heterogeneity factor
PFO – pseudo first order
PSO – pseudo second order
t – time
q_t – amount of adsorbate adsorbed at time t (mg/g)
q_e – amount of adsorbate adsorbed at equilibrium (mg/g)
k_1 – PFO rate constant (min^{-1})
k_2 – PSO rate constant ($\mathrm{g/mg \cdot min}$)
R^2 – coefficient of determination

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