

Physicochemical characteristics and nutrient-specific net retention and reuse of rice husk biochar for aquaculture water quality management

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Abstract. Rice husk biochar may support aquaculture water-quality management, but its performance depends on nutrient retention, release, and reuse. This study characterized rice husk biochar and evaluated total ammonia nitrogen (TAN), nitrate, and phosphate retention and desorption under simultaneous-contact conditions. Biochar was produced by slow pyrolysis at 450°C for 2 h. Characterization covered physicochemical properties, surface morphology, functional groups, elemental composition, and surface area. Experiments used 1 g of biochar in 150 mL of a multi-nutrient solution, with biochar-free controls and three units per sampling time. Net retention was calculated from concentration differences between treatment and control. The biochar had an alkaline pH (8.9), high ash content (42.72%), a surface area of 8.81 m^2 g^{-1}, and a point of zero charge of approximately 8.03. At 216 h, apparent net TAN retention was positive during the first two cycles but negative during the third, indicating net release relative to the control. Nitrate showed fluctuating retention and readsorption, although apparent net retention was positive at the selected comparison time in both phases. Phosphate retention was limited and inconsistent, while leaching and desorption indicated that the biochar could release phosphate. Its available phosphorus content (336.51 mg kg^{-1}) may have contributed. These findings show that rice husk biochar is not a universal nutrient adsorbent. Aquaculture applications require nutrient-specific assessment of retention, release, and reuse, alongside evaluation of alkalinity contribution and pH-buffering capacity under actual pond conditions.

Keywords: ammonia nitrogen, desorption, nutrient leaching, nutrient sorption, water quality.

Introduction. Aquaculture is one of the fastest-growing food-production sectors and contributes substantially to global animal-protein supply. However, increasing production intensity and anthropogenic pressures in coastal areas and watersheds can degrade the quality of source water used for aquaculture. Agricultural runoff, domestic wastewater, industrial activities, and accumulated waste from previous production cycles can increase nitrogen and phosphorus inputs to receiving waters (Adyasari et al., 2021). Consequently, source water may already be degraded before entering aquaculture systems, increasing the risk of water-quality deterioration during production (Boyd & Tucker, 1998; FAO, 2022).

Total ammonia nitrogen (TAN) is a critical water-quality parameter because the un-ionized fraction of ammonia (NH_3) is toxic to aquatic organisms, and its proportion increases with pH and temperature. Elevated TAN can impair growth, metabolism, and respiration and increase disease susceptibility and mortality when concentrations exceed species-specific tolerance limits (Wicks et al., 1999; Colt, 2006). Nitrate (NO_3^-) and phosphate (PO_4^{3-}) are also important because their accumulation can contribute to eutrophication, excessive phytoplankton growth, dissolved-oxygen fluctuations, and reduced ecosystem stability (Boyd & Tucker, 1998; Camargo & Alonso, 2006).

Conventional nutrient-control approaches include water exchange, biological filtration, zeolite, and synthetic adsorbents. These approaches can involve relatively high operating costs, maintenance requirements, and declining material performance during repeated use (Crini & Lichtfouse, 2019). Biochar is therefore of interest as a low-cost, biomass-derived material for water-quality management (Sawant et al., 2024). Rice husk is particularly attractive in rice-producing regions such as Indonesia because it is abundant and can be converted into a carbon-rich material within a circular-bioeconomy framework (Lehmann & Joseph, 2009; Mohan et al., 2014).

Most biochar studies emphasize initial nutrient removal or adsorption capacity. For practical water-quality management, however, performance also depends on whether retained nutrients can subsequently be released under changing conditions and whether the material retains nutrients again after reuse (Zhang et al., 2020; Alsawy et al., 2022). Information on this retention–release–reuse behavior remains limited for rice husk biochar, particularly under simultaneous exposure to multiple dissolved nutrients.

Different nutrients may also interact differently with biochar. NH_4^+ may interact with negatively charged surface sites and cation-exchange sites, whereas nitrate and phosphate are anions whose retention can be influenced by surface charge, mineral phases, pH, and other physicochemical properties of the biochar (Inyang et al., 2010; Tan et al., 2015).

Nutrient-specific evaluation is necessary to understand how biochar properties relate to the retention and release of different nutrient species. In this study, rice husk biochar was characterized, and its net retention, release, and reuse behavior for TAN, nitrate, and phosphate was evaluated under simultaneous-contact conditions. Therefore, the objective was to characterize these nutrient-specific responses and to evaluate the potential of rice husk biochar to regulate dissolved nutrient availability in aquaculture water.

Material and Method

Study period and experimental site. The study was conducted from January to July 2026 at the laboratory of the Center Aquaculture Production Business, Ministry of Marine Affairs and Fisheries of the Republic of Indonesia, Cilebar District, Karawang Regency, West Java, Indonesia.

Rice (*Oryza sativa* L.) husk was obtained from a rice mill in Cilebar District, Karawang Regency, West Java, Indonesia. The material was cleaned and oven-dried at 105°C for 24 h to constant mass.

Pyrolysis was conducted using three separate closed steel reactors (1.8-mm wall thickness; 15 × 10 × 6 cm), each equipped with a tight lid and a small vent to allow pyrolysis gases to escape while limiting oxygen entry. The three reactors were operated under the same pyrolysis conditions. The reactors were heated in a muffle furnace (Thermo Scientific Barnstead/Thermolyne, USA) to 450°C at approximately 16°C min^{-1} and held for 2 h. The furnace was then switched off, and the reactors were allowed to cool to room temperature.

Biochar produced from the three reactors was subsequently combined to form a bulk biochar stock for subsequent characterization and nutrient retention, release, and reuse experiments. The biochar was retained in its coarse, unground rice-husk form to represent the material used under practical application conditions. The bulk biochar was stored in an airtight container at room temperature until further analysis.

Biochar characterization. The biochar was characterized for physical, chemical, surface, and morphological properties relevant to nutrient interactions. The parameters included pyrolysis yield, pH, point of zero charge (pH_pzc), moisture, volatile matter, ash, fixed carbon, bulk density, total nitrogen, available phosphorus, cation-exchange capacity (CEC), surface functional groups, elemental composition, surface morphology, specific surface area, and pore characteristics.

Biochar yield was calculated as the ratio of dry biochar mass after pyrolysis to dry feedstock mass before pyrolysis. Biochar pH was measured in water at a 1:5 biochar-to-

solution ratio. Total nitrogen by wet digestion followed by titration, available phosphorus by the Olsen method, and CEC by ammonium-acetate percolation followed by titration. Moisture, volatile matter, and ash were determined by proximate analysis, while fixed carbon was calculated by difference. Bulk density was determined by measuring the mass of biochar occupying a known volume and calculating the mass-to-volume ratio.

The pH_pzc was determined using the pH-drift method. A series of electrolyte solutions with different initial pH values was prepared, and a fixed mass of biochar was added to each solution. After equilibration, the final pH was measured. The pH_pzc was determined from the relationship between the initial and final pH values at which the net change in pH approached zero.

Surface functional groups were examined by Fourier-transform infrared spectroscopy (FTIR) over 4000-400 cm^{-1}. Surface morphology and elemental composition were examined by scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS/EDX). Specific surface area and pore characteristics were determined by the Brunauer–Emmett–Teller (BET) method using nitrogen adsorption–desorption isotherms.

Nutrient retention, release and reuse. Retention, release, and reuse experiments were conducted for TAN, nitrate, and phosphate under simultaneous-contact conditions. Before the nutrient experiments, the biochar was washed with 1 M HCl to reduce soluble ash and mineral impurities. The material was then neutralized with $NaHCO_3$ to approximately neutral pH, rinsed with distilled water, filtered, and oven-dried at 65°C for 24 h. Stock solutions were prepared using analytical-grade NH_4Cl, KNO_3, and $NH_4H_2PO_4$. Theoretical nutrient concentrations were calculated stoichiometrically, whereas all experimental calculations were based on measured nutrient concentrations.

Retention experiments were conducted using 1 g of dry biochar in 150 mL of multi-nutrient solution in 250-mL plastic bottles. The bottles were closed and maintained at room temperature. A biochar-free control solution was prepared and monitored in parallel to account for concentration changes unrelated to biochar, including potential precipitation, transformation, or analytical variation.

A sufficient number of reaction bottles was prepared at the beginning of the experiment to allow destructive sampling at the scheduled observation times. At each sampling time, three separate reaction bottles were collected for analysis. Each sampling time therefore represented three experimental units (n = 3), and the sampled bottles were not returned to the experiment.

Net nutrient retention at time *t* was calculated relative to the corresponding control concentration:

$$R_t\,(\%) = (C_{control,t} - C_{biochar,t}) \times 100/C_{control,t}$$

where R_t is net nutrient retention at time *t* (%), $C_{control,t}$ is the nutrient concentration in the control solution at time *t* (mg L^{-1}), and $C_{biochar,t}$ is the nutrient concentration in the biochar-treated solution at the same time (mg L^{-1}).

Positive values indicate a lower nutrient concentration in the biochar-treated solution than in the control, whereas negative values indicate a higher concentration in the biochar-treated solution and were therefore interpreted as net nutrient release.

The apparent net nutrient retention per unit mass of biochar was calculated as:

$$q_t = (C_{control,t} - C_{biochar,t}) \times V/m$$

where q_t is the apparent net nutrient retention at time *t* (mg g^{-1}), V is the solution volume (L), and m is the dry biochar mass (g). Because q_t was calculated from the difference between the biochar-treated and control solutions, it was interpreted as apparent net retention per unit mass, rather than as an equilibrium adsorption capacity.

Following the defined retention period, the biochar was separated from the solution by filtration and briefly rinsed with distilled water before the release experiment. The recovered biochar was then contacted with 150 mL of distilled water. Released TAN, nitrate, and phosphate were measured at the scheduled sampling times. Release was quantified from the measured nutrient concentration in the release medium.

Three separate experimental units were analyzed at each release sampling time. Each unit was sampled only once; therefore, the release experiment also followed a destructive-sampling design.

Following the release experiment, the recovered biochar was used in a reuse cycle by contacting it with a fresh 150-mL multi-nutrient solution under the same experimental conditions as the initial retention experiment.

Reuse performance was evaluated using net nutrient retention relative to the corresponding time-matched control:

$$R_{reuse,t}\ (\%) = (C_{control,t} - C_{reuse,t}) \times 100/C_{control,t}$$

where $R_{reuse,t}$ is net nutrient retention during reuse at time *t* (%), $C_{control,t}$ is the nutrient concentration in the corresponding control solution (mg L^{-1}), and $C_{reuse,t}$ is the nutrient concentration in the solution containing reused biochar (mg L^{-1}). A second reuse cycle was not performed for phosphate.

Data analysis. All nutrient retention, release, and reuse experiments were conducted with three experimental units at each sampling time (n = 3). Results were expressed as mean±standard deviation (SD). Data were analyzed descriptively to characterize temporal patterns of nutrient-specific net retention, release, and reuse. For each sampling time, the mean and sample standard deviation were calculated from the three experimental units.

For descriptive comparison among contact cycles, mean net retention was calculated as the arithmetic mean of the time-specific mean net-retention values across the observation times defined for each cycle. Cycle-level mean values were treated as descriptive summaries and were not considered independent statistical replicates. Negative net-retention values were retained in all calculations because they represent net nutrient release relative to the control. All calculations, descriptive statistics, and figures were prepared using Microsoft Excel.

Results

Physicochemical characteristics of rice husk biochar. The physicochemical properties of the rice husk biochar are summarized in Table 1. The material exhibited an alkaline pH, a relatively high ash content, and a substantial fixed-carbon fraction. Its measured surface area and pore characteristics are also presented in Table 1.

EDX analysis showed that oxygen and silicon were the dominant detected surface elements. SEM revealed an irregular, heterogeneous surface with cavities of different sizes (Figure 1).

Table 1

Physicochemical characteristics of rice husk biochar

Category	*Parameter*	*Value*	*Unit*
Physical	Pyrolysis yield	42.6	%
Physical	Bulk density	0.39	g cm^{-3}
Physical	Specific surface area	8.81	m^2 g^{-1}
Physical	Average pore diameter	10.51	nm
Chemical	pH	8.9	—
Chemical	Volatile matter	16.36	%
Chemical	Ash	42.72	%
Chemical	Fixed carbon	40.92	%
Chemical	Total nitrogen	0.39	%
Chemical	Available phosphorus	336.51	mg kg^{-1}
Chemical	CEC	12.42	cmol(+) kg^{-1}
EDX	O	60.38	wt.%
EDX	Si	26.98	wt.%
EDX	K	0.44	wt.%

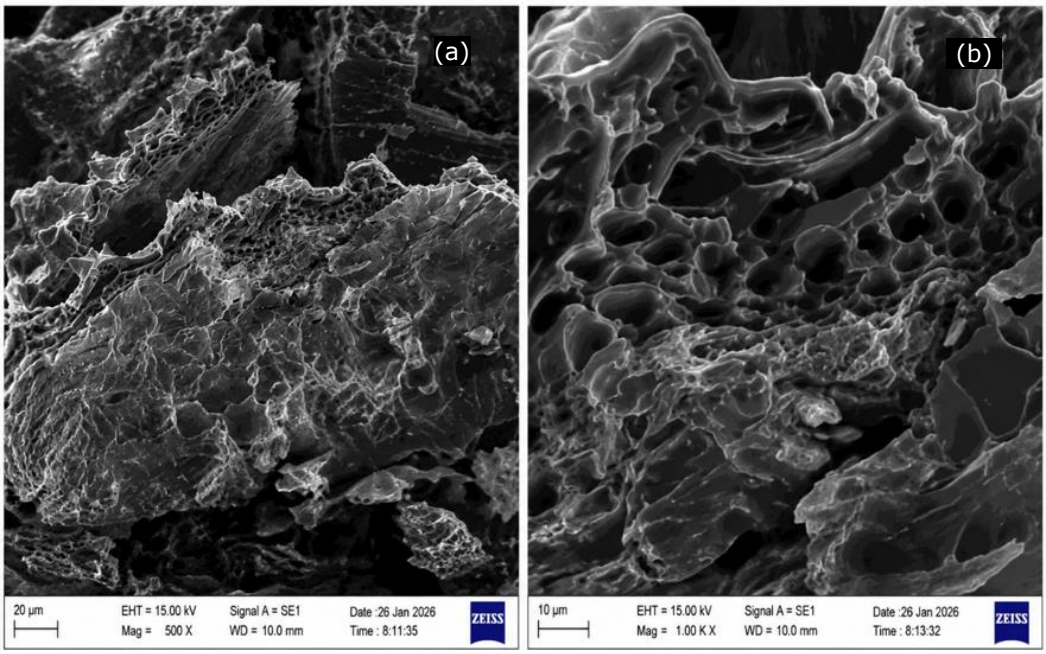


Figure 1. Surface morphology of rice husk biochar by scanning electron microscopy at (a) 500× and (b) 1000× magnification.

The FTIR spectrum showed bands associated with hydroxyl, aliphatic, carbonyl/aromatic, and silicate-related structures (Figure 2). The band at 3301.31 cm^{-1} was assigned primarily to O–H stretching, indicating hydroxyl-containing groups. The band at 2898.17 cm^{-1} corresponded to aliphatic C–H stretching. The band at 1794.84 cm^{-1} was tentatively attributed to carbonyl (C=O) stretching, while the band at 1642.46 cm^{-1} was associated with aromatic C=C structures and/or oxygen-containing carbonyl groups. The band at 1446.67 cm^{-1} was associated with C–H deformation and/or aromatic structures. The band at 1156.37 cm^{-1} was attributed to C–O–C and/or C–O vibrations, whereas the strong band at 1030.83 cm^{-1} was associated with C–O and/or Si–O–Si vibrations. Finally, the band at 878.65 cm^{-1} was tentatively assigned to out-of-plane aromatic C–H deformation and/or silicate-related vibrations. Overall, the spectrum indicated the presence of hydroxyl-containing groups, aliphatic and aromatic structures, oxygen-containing functionalities, and silica-related mineral components in the rice husk biochar.

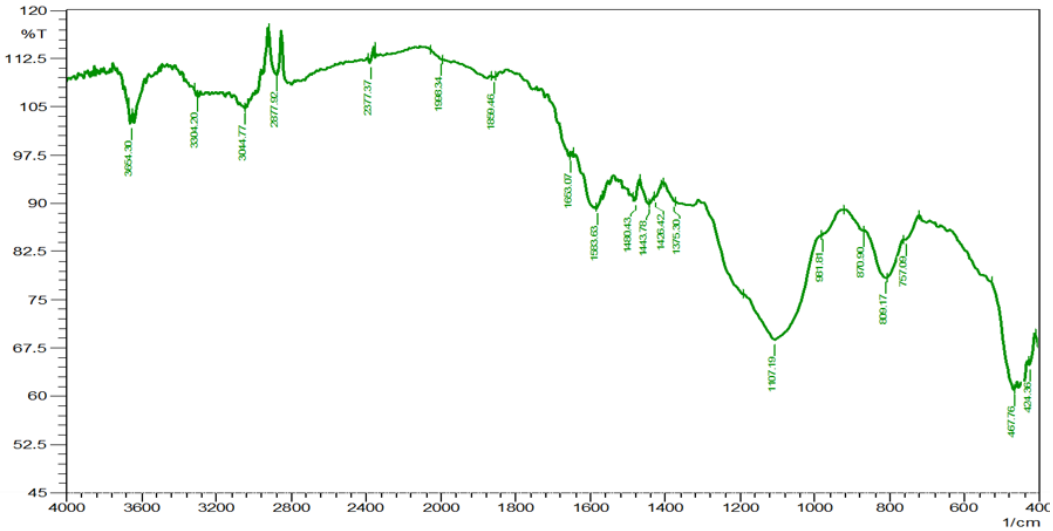


Figure 2. FTIR spectrum of rice husk biochar produced at 450°C.

The pH-drift experiment showed that changes in solution pH depended on the initial pH. The largest positive ΔpH occurred at an initial pH of 4, whereas ΔpH was close to zero at an initial pH of 8. The estimated pH_pzc was approximately 8.03 (Figure 3).

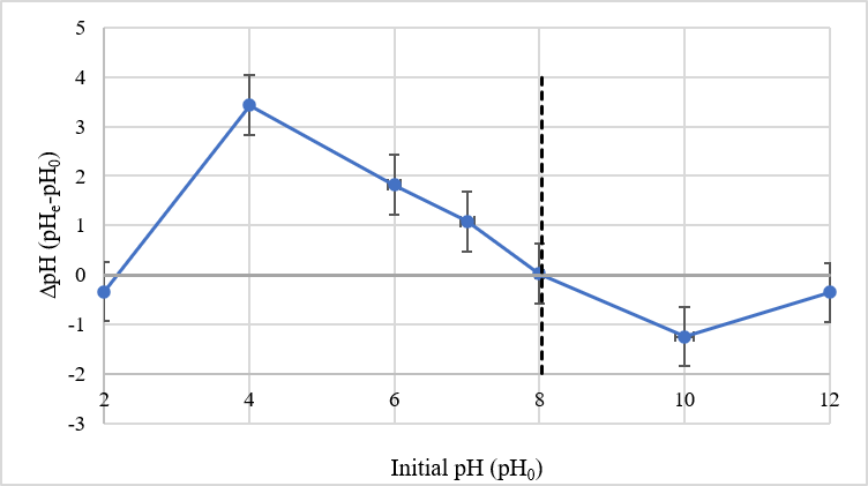


Figure 3. pH-drift profile of rice husk biochar and determination of its point of zero charge (pH_pzc) after 24 h of equilibration.

Initial nutrient release from untreated biochar. The initial leaching test showed different release patterns among the three nutrients (Table 2).

Table 2

Nutrient release from untreated rice husk biochar during a 21-day leaching test

Nutrient	*Day 7 (mg L^{-1})*	*Day 14 (mg L^{-1})*	*Day 21 (mg L^{-1})*	*Apparent amount released at day 21 (mg g^{-1})*
TAN	0.69	0.76	0.91	0.14
NO_3^-	0.23	ND	0.41	0.06
PO_4^{3-}	1.88	0.26	2.32	0.35

Note: ND, not detected.

TAN concentrations increased across the observation days, whereas nitrate was not detected on day 14 but was measurable on days 7 and 21. Phosphate exhibited the largest concentration fluctuation, with a marked decrease on day 14 followed by an increase on day 21. Among the nutrients examined, phosphate had the highest measured concentration at the final sampling time.

TAN retention, desorption, and reuse across successive cycles. Net TAN retention varied across the three successive biochar contact cycles (Figure 4). During the first and second cycles, net retention increased toward the later contact period, whereas the third cycle exhibited a contrasting pattern, with net retention becoming negative at the final observation. These differences indicate that the net retention behavior of TAN was not consistent across successive cycles.

TAN release during desorption also changed over time. In the first and second cycles, the amount of TAN released increased during the later stages of desorption. This pattern indicates that TAN retention in the biochar-solution system was at least partly reversible under the experimental conditions.

At the common comparison time of 216 h, the apparent net TAN retention capacity (qt) was 0.24, 0.67, and −0.42 mg g^{-1} for cycles I, II, and III, respectively. The corresponding net retention percentages were 10.99%, 20.44%, and −13.36%. Thus, net TAN retention was positive in the first two cycles but negative in the third cycle, indicating net TAN release relative to the control during the third cycle. These findings demonstrate cycle-dependent changes in the net retention behavior of TAN during biochar reuse.

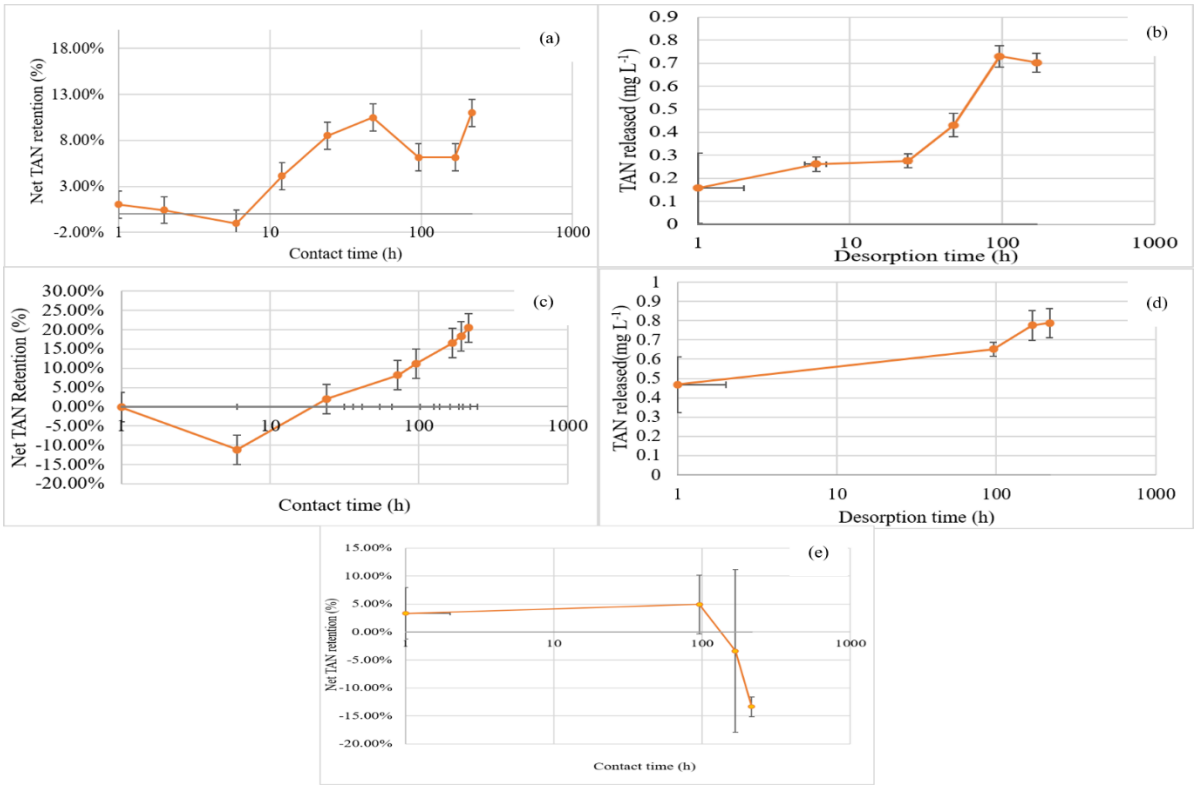


Figure 4. Time-dependent net TAN retention and desorption behavior of rice husk biochar during successive contact cycles. Panels (a), (c), and (e) show net TAN retention, whereas panels (b) and (d) show TAN release during desorption.

Nitrate retention, desorption, and reuse across successive cycles. Net nitrate retention during the first contact cycle fluctuated throughout the observation period, with both positive and negative values (Figure 5(a)). This indicates that net nitrate retention relative to the control was not consistent over time. During desorption, nitrate concentrations also varied across sampling times, reaching higher values at intermediate observations before declining to low levels at the end of the observation period (Figure 5(b)).

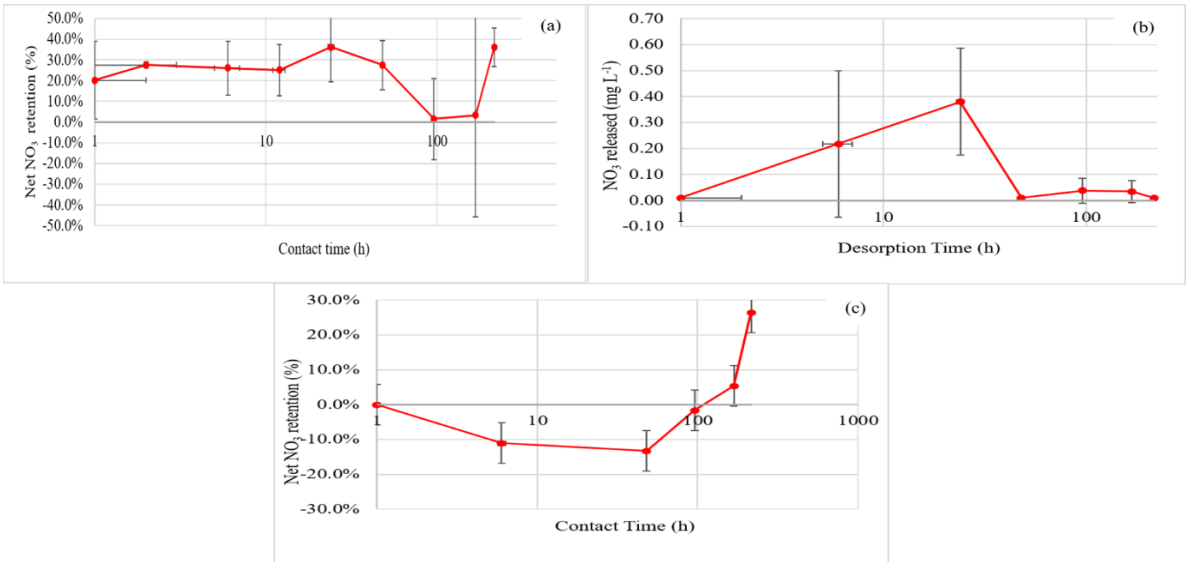


Figure 5. Nitrate retention, desorption, and readsorption during the first biochar contact cycle. (a) Net nitrate retention during the initial contact phase (%); (b) nitrate released during desorption (mg L^{-1}); and (c) net nitrate retention during the readsorption phase (%).

During the subsequent readsorption phase, net nitrate retention again fluctuated, initially showing negative values before becoming positive toward the end of the contact period (Figure 5(c)). At the selected comparison time of 216 h, the apparent net nitrate retention capacity (qt) was 0.12 mg g^{-1} during the initial retention phase and 0.13 mg g^{-1} during readsorption. These results indicate net nitrate retention relative to the corresponding control in both phases at the selected comparison time, despite the fluctuating patterns observed throughout the contact period.

Phosphate retention and desorption. Net phosphate retention during the first contact cycle fluctuated throughout the observation period, with both positive and negative values observed (Figure 6(a)). The retention response did not follow a consistent trend over time. At the selected comparison time of 216 h, the apparent net phosphate retention capacity (qt) was 0.00370 mg g^{-1}, indicating a small net difference between the control and biochar treatment.

Phosphate concentrations in the desorption solution also varied across the observation period (Figure 6(b)). At 216 h, phosphate was not detected in any of the three replicates. This indicates that the phosphate concentration in the desorption solution was below the detection threshold of the analytical method at the final observation time.

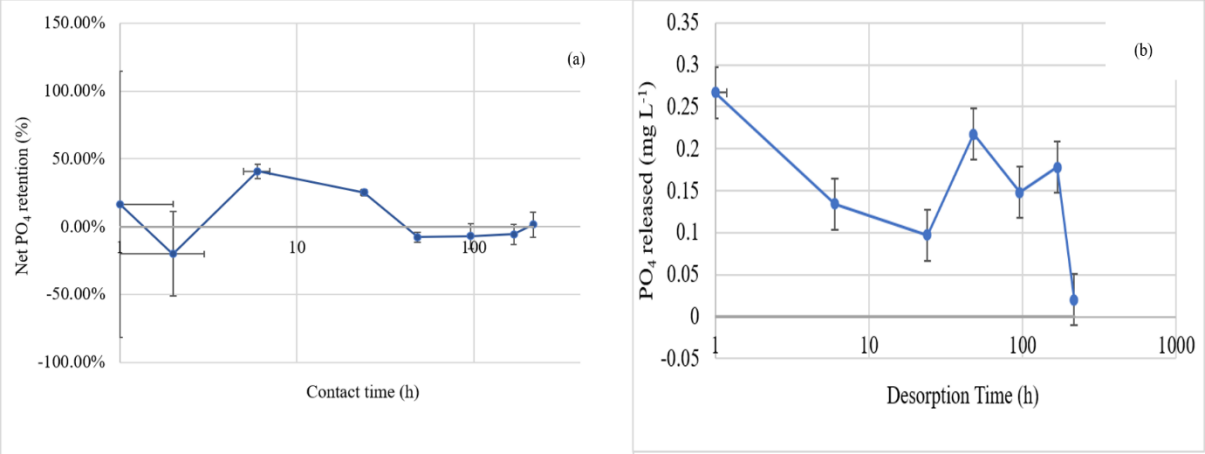


Figure 6. Phosphate retention and desorption during the first biochar contact cycle. (a) Net phosphate retention (%); (b) phosphate released during desorption (mg L^{-1}).

Discussion

Physicochemical properties of rice husk biochar and their implications for nutrient interactions. The physicochemical properties of rice husk biochar provide an important basis for interpreting its nutrient-specific retention and release behavior. In the present study, the biochar exhibited an alkaline pH (8.9), a high ash content (42.72%), a BET surface area of 8.81 m^2 g^{-1}, and a CEC of 12.42 cmol(+) kg^{-1}. These characteristics indicate that nutrient interactions may involve several mechanisms, including ion exchange, surface interactions, and reactions involving mineral constituents, rather than being governed exclusively by surface area. Biochar properties are strongly influenced by feedstock composition and pyrolysis conditions, which determine the resulting pore structure, surface chemistry, mineral content, and ion-exchange characteristics (Tomczyk et al., 2020).

The alkaline pH and high ash content suggest that mineral constituents may contribute to acid-base interactions when the biochar contacts water. During pyrolysis, the relative concentration of inorganic constituents can increase as organic matter decomposes, and alkaline mineral phases may contribute to the pH of biochar suspensions (Tomczyk et al., 2020). Nevertheless, ash content alone does not quantify alkalinity, and the contribution of specific minerals cannot be established from the present characterization data. The measured pH should therefore be interpreted as a

property of the material under the test conditions, rather than as direct evidence of its capacity to maintain a particular pH in aquaculture water.

The measured pH_pzc of approximately 8.03 provides additional information about the potential surface-charge behavior of the biochar. In general, a surface tends to become positively charged below its pH_pzc and negatively charged above it, although the magnitude of the charge also depends on surface functional groups, ionic strength, and solution chemistry (Kasera et al., 2022). This distinction is relevant because TAN, which includes ammonium and ammonia, and the anionic forms of nitrate and phosphate differ in their interactions with charged surfaces. At a solution pH below the pH_pzc, protonated surface sites may favor electrostatic attraction of anions, whereas negatively charged sites may favor cation interactions at pH values above the pH_pzc. These principles provide a mechanistic framework, but they do not establish the actual surface charge or dominant adsorption mechanism in the nutrient solutions used in this study.

The BET surface area of 8.81 $m^2\ g^{-1}$ was relatively low compared with highly porous or activated biochar. However, adsorption performance cannot be predicted from surface area alone, because pore-size distribution, pore accessibility, surface functional groups, and the physicochemical properties of the target ions also influence sorption (Ahmad et al., 2014; Leng et al., 2021). This is particularly relevant to rice husk biochar, whose adsorption properties can vary with pyrolysis conditions and post-treatment. For example, Mathurasa & Damrongsiri (2018) demonstrated that modification of rice husk-derived materials altered surface charge and adsorption performance for ammonium and nitrate.

The measured CEC of 12.42 $cmol(+)\ kg^{-1}$ may be relevant to ammonium retention because NH_4^+ can interact with negatively charged exchange sites. Nevertheless, CEC is an indicator of potential cation-exchange capacity, not a direct measurement of ammonium adsorption under the conditions of the present experiment. Its actual contribution would depend on solution pH, competing ions, and the accessibility and chemical characteristics of exchange sites. In contrast, nitrate and phosphate are anions under the relevant aqueous conditions, so their interactions with biochar cannot be inferred from CEC alone. Mathurasa & Damrongsiri (2018) showed that changes in surface charge through material modification affected nitrate adsorption, highlighting the importance of surface chemistry when considering anionic nutrients.

The irregular surface morphology and cavities observed by SEM indicate structural heterogeneity in the biochar. However, SEM images alone cannot establish whether these cavities were accessible to dissolved nutrients or whether they acted as effective adsorption sites. As discussed by Leng et al. (2021), pore structure and surface functional groups both influence adsorption, and their contributions depend on the adsorbate and the properties of the biochar. Accordingly, the SEM observations in this study are best interpreted as complementary morphological evidence, rather than direct proof of the mechanism responsible for nutrient retention.

The FTIR spectrum showed bands tentatively assigned to hydroxyl (O–H), carbonyl-related (C=O), and C–O-containing groups, as well as vibrations potentially associated with silicate structures. Oxygen-containing functional groups can influence the surface chemistry of biochar and its interactions with dissolved substances; however, the contribution of individual groups depends on their chemical environment and the properties of the solution (Ahmad et al., 2014; Tomczyk et al., 2020). FTIR band assignments should be interpreted cautiously because different functional groups may produce overlapping absorption bands (Coates, 2006). Therefore, the observed bands indicate possible functional groups but do not, by themselves, establish which groups were responsible for nutrient retention in the present study.

The elemental analysis detected substantial oxygen and silicon signals, suggesting that the material contained both carbonaceous and mineral-associated components. This interpretation is consistent with the composition of rice husk-derived biochar, in which silica and other inorganic constituents may remain after pyrolysis (Tomczyk et al., 2020). Nevertheless, elemental analysis by EDX does not identify specific mineral phases or determine the chemical bonding of each element. Consequently, the combined FTIR and EDX results provide complementary evidence of the material's surface chemistry and

elemental composition, but further analyses would be required to identify specific mineral phases or demonstrate a direct relationship between individual functional groups and nutrient retention.

Importantly, the relevance of these properties differs among nutrients. Pratiwi et al. (2016) reported that rice husk char retained ammonium and nitrate in batch experiments but showed little phosphate sorption and could increase phosphate leaching from loamy soil. Mathurasa & Damrongsiri (2018) likewise demonstrated that the adsorption of ammonium and nitrate by rice husk-derived materials depended on material preparation and surface modification. These findings indicate that rice husk biochar should not be treated as a universal nutrient adsorbent. Instead, its performance needs to be evaluated separately for each nutrient and under the specific solution conditions applied.

The initial leaching test in the present study showed that untreated rice husk biochar released TAN, nitrate, and phosphate, although their temporal patterns differed. Nutrient-specific retention and release have also been reported by Pratiwi et al. (2016), who found that rice husk char retained ammonium and nitrate but exhibited little phosphate sorption and released phosphate into solution under certain concentration conditions. These findings indicate that biochar–nutrient interactions may involve both retention and release rather than adsorption alone. Accordingly, changes in nutrient concentrations in the present contact experiments are interpreted as net concentration changes relative to the corresponding controls, rather than as direct measurements of adsorption alone.

Nutrient-specific net retention and release behavior. The results demonstrate that the net retention and release behavior of rice husk biochar differed among TAN, nitrate, and phosphate. This nutrient-specific response is consistent with the review by Zhang et al. (2020), which showed that the retention of ammonium, nitrate, and phosphate by biochar depends on their chemical properties, biochar surface characteristics, solution pH, and the presence of competing ions. In particular, ammonium retention may involve cation exchange and interactions with oxygen-containing functional groups, whereas nitrate retention can be limited by electrostatic repulsion from negatively charged surfaces. Phosphate retention, in turn, may depend strongly on interactions with mineral components, particularly metal oxides. These differences provide a useful framework for interpreting the distinct responses observed in the present study, although the dominant mechanisms were not directly determined.

For TAN, the net retention response varied across successive contact cycles. At the selected comparison time of 216 h, apparent net retention was positive during the first and second cycles but negative during the third cycle. The negative value indicates that TAN concentration in the biochar treatment exceeded that in the corresponding control, representing net release relative to the control rather than retention. This pattern is consistent with the broader observation that biochar can both retain nutrients and release endogenous nutrients into water, depending on material properties and experimental conditions (Zhang et al. 2020). However, the increase in apparent net retention between the first and second cycles should not be interpreted as evidence that reuse improved the intrinsic adsorption capacity of the biochar. Differences in control concentrations, solution chemistry, and the history of the material may have contributed to the observed changes.

Nitrate showed a fluctuating net retention pattern during both the initial contact and subsequent readsorption phases. Positive and negative net retention values indicate that nitrate concentrations in the biochar treatment were sometimes lower and sometimes higher than those in the corresponding controls. Pratiwi et al. (2016) reported that rice husk char could retain nitrate under their batch experimental conditions, but also demonstrated that nutrient behavior depended on the conditions under which retention and leaching were assessed. Zhang et al. (2020) further noted that nitrate retention by unmodified biochar is often limited by electrostatic repulsion from negatively charged surfaces. These studies help explain why nitrate retention should not be assumed to remain consistently positive throughout an experiment. Nevertheless, the present data do not establish whether the observed fluctuations resulted from surface-charge effects,

release from the material, variation among experimental units, or changes in solution chemistry.

Phosphate exhibited a different response, with fluctuating net retention and a small apparent net retention value at the selected comparison time. Phosphate was also not detected in the desorption solution at the final observation. These observations should be interpreted separately: the small net retention value reflects the difference between phosphate concentrations in the treatment and control at that time, whereas the nondetection during desorption indicates that the measured concentration was below the analytical detection limit. Neither observation alone proves that phosphate was not retained by the biochar. In a study specifically examining rice husk char, Pratiwi et al. (2016) reported little phosphate sorption and observed phosphate release under certain solution conditions. This provides a relevant comparison with the present findings and highlights the possibility that rice husk biochar may behave as a phosphate source rather than an effective phosphate-retention material under some conditions. However, differences in experimental design, initial nutrient concentrations, and solution chemistry prevent direct quantitative comparison between the two studies.

An important consideration is that the present study evaluated net concentration changes relative to time-matched biochar-free controls, rather than measuring adsorption independently of release. Accordingly, positive net retention values indicate lower nutrient concentrations in the biochar treatment than in the corresponding control, whereas negative values indicate higher concentrations in the treatment. This approach captures the overall effect of biochar contact under the experimental conditions, including the possibility of simultaneous retention and release. Zhang et al. (2020) similarly identified the release of endogenous nitrogen and phosphorus as a potential limitation of biochar-based nutrient removal. Therefore, interpreting the results solely as adsorption capacity would overlook the dynamic nature of the observed responses.

Overall, the findings indicate that rice husk biochar did not exhibit a uniform retention response across the three nutrients or across successive contact cycles. TAN and nitrate showed cycle- or time-dependent changes in net retention, whereas phosphate showed limited net retention at the selected comparison time and was not detected in the final desorption sample. These results support nutrient-specific evaluation of rice husk biochar rather than assuming that it will consistently remove all dissolved nutrients. Because the experiments were conducted under controlled laboratory conditions, further testing in aquaculture water and under operational pond conditions is required before drawing conclusions about its practical effectiveness in aquaculture water-quality management.

Desorption and retention behavior during biochar reuse*.* The results indicate that the net retention behavior of rice husk biochar changed across successive contact cycles, suggesting that its performance during reuse was not constant. Regeneration and reuse are important considerations for adsorbent applications because the effectiveness of a material may depend on the reversibility of nutrient interactions, the accessibility of active sites, and changes in surface properties during repeated use (Alsawy et al., 2022). However, the present experiment did not independently quantify changes in active sites or establish the mechanisms responsible for the observed cycle-dependent responses.

For TAN, apparent net retention at the selected comparison time was positive during the first two contact cycles but negative during the third. The negative value indicates that the TAN concentration in the biochar treatment exceeded that in the corresponding control. This result is important because it shows that reuse did not consistently produce lower TAN concentrations relative to the control. One possible interpretation is that the balance between retention and release changed during successive cycles; however, the data do not establish whether this change resulted from altered surface properties, nutrient release from the biochar, or differences in solution conditions. Zhang et al. (2020) noted that endogenous nitrogen release can limit the effectiveness of biochar for nutrient removal, supporting the need to consider release alongside apparent retention when evaluating biochar performance.

Nitrate also exhibited variable behavior during the initial contact and subsequent readsorption phases. At the selected comparison time, apparent net nitrate retention was positive in both phases, but the time-course data showed fluctuations rather than a consistently increasing retention response. This indicates that a positive result at one comparison time does not necessarily represent stable performance throughout a contact cycle. The review by Zhang et al. (2020) explains that nitrate retention by unmodified biochar can be limited by electrostatic repulsion from negatively charged surfaces, although the actual behavior depends on the material and solution conditions. This provides a possible context for interpreting the observed variability, but electrostatic repulsion was not directly tested in the present experiment.

Previous studies have shown that biochar can retain adsorption performance over multiple regeneration cycles, although the outcome depends on the biochar feedstock, target compound, and regeneration procedure. For example, Alsawy et al. (2022) reviewed how regeneration efficiency is influenced by adsorption mechanisms, functional groups, pore structure, and the regeneration conditions. Their review also emphasizes that regeneration does not necessarily restore the original material completely. The present experiment differs from conventional regeneration studies because biochar was exposed to nutrient solution, transferred to distilled water for desorption, and then reused in fresh nutrient solution. Therefore, the observed responses should be described as changes in net retention and release during reuse, rather than as a definitive assessment of regeneration efficiency. Overall, the TAN and nitrate results show that the behavior of rice husk biochar varied during successive use, and that reuse did not guarantee consistently positive net retention.

Phosphate was not evaluated in subsequent reuse cycles because the initial retention test showed limited and inconsistent net retention, while phosphate was detected in the desorption solution. This response suggests that, under the experimental conditions, rice husk biochar did not function consistently as a net phosphate-retention material and could also act as a source of dissolved phosphate. The relatively high available phosphorus content measured in the biochar (336.51 mg kg^{-1}) provides a plausible explanation for this release, although the contribution of the biochar's internal phosphorus pool was not directly quantified. Therefore, subsequent reuse tests were restricted to TAN and nitrate, for which evaluating changes in net retention across successive cycles was considered more informative. This decision should not be interpreted as evidence that rice husk biochar cannot retain phosphate under other conditions; rather, it reflects the nutrient-specific behavior observed in the present experiment.

Implications for aquaculture water quality management and study limitations. The findings have several implications for the potential use of rice husk biochar in aquaculture water-quality management. First, the different responses observed for TAN, nitrate, and phosphate indicate that biochar should not be assumed to remove all dissolved nutrients simultaneously. The nutrient-removal performance of biochar depends on its physicochemical properties, the chemical form of the target nutrient, solution pH, and the presence of competing ions (Zhang et al., 2020). Furthermore, biochar may release endogenous nitrogen and phosphorus, which can offset its apparent nutrient-removal performance. Consequently, its suitability for aquaculture applications should be evaluated by considering both nutrient retention and nutrient release.

Second, rice husk biochar may contribute to alkalinity supply and influence pH regulation in aquaculture water. Biochar commonly contains alkaline mineral constituents, and its acid–base properties are influenced by feedstock composition and pyrolysis conditions (Tomczyk et al., 2020; Karam et al., 2022). In the present study, the biochar exhibited an alkaline pH of 8.9 and a high ash content of 42.72%. In addition, the 24-h pH-drift experiment yielded an estimated (pH_pzc of approximately 8.03. The observed pH-drift pattern indicates that the interaction between the biochar surface and the surrounding solution was pH-dependent, with the final pH approaching a value near the estimated pH_pzc under the test conditions. This provides empirical evidence of acid–base interactions between the biochar and the solution and supports the potential role of its

surface chemistry in influencing pH changes. However, pH_pzc represents the pH at which the net surface charge is approximately zero; it is not, by itself, a direct measurement of alkalinity or buffering capacity. The alkaline pH and high ash content suggest that mineral constituents may also contribute to acidity neutralization, but their specific contribution was not quantified. Thus, the pH-drift results support the potential of rice husk biochar to influence solution pH, while its capacity to supply alkalinity and resist pH fluctuations requires further quantitative evaluation. This distinction is important because alkalinity supply refers to the ability to neutralize acidity, whereas buffering capacity describes resistance to pH changes when acid or base is added.

Third, the biochar may act as a source of dissolved phosphate as well as a material capable of retaining phosphate under some conditions. Ghodszad et al. (2021) reported that biochar can affect phosphorus availability through both release and retention processes, with the direction and magnitude of the response depending on feedstock properties, pyrolysis conditions, and solution chemistry. In the present study, the biochar contained 336.51 mg kg^{-1} of available phosphorus, and phosphate was measured in the initial leaching test and in the desorption solution. These findings suggest that the biochar's internal phosphorus pool may have contributed to phosphate release, although the contribution of this pool was not independently quantified. The relatively low and fluctuating net phosphate retention observed in the retention experiment further indicates that this material did not consistently function as a net phosphate-retention medium under the tested conditions. Therefore, its potential role as a phosphorus source should be distinguished from its effectiveness as a phosphate-removal material. In aquaculture systems, released phosphate could contribute to nutrient availability and primary production, but excessive nutrient availability may also increase the risk of undesirable algal proliferation. These possible consequences were not directly tested in the present experiment and require validation under pond conditions.

The results also highlight the importance of evaluating biochar performance over time and during repeated use. Positive net retention at a particular sampling time does not demonstrate that retention will remain positive throughout a contact period or across subsequent cycles. The negative net retention observed for TAN during the third cycle and the fluctuating nitrate responses illustrate this limitation. Because net retention was calculated relative to the corresponding controls, the resulting values represent differences in nutrient concentrations between the biochar treatment and the control, rather than direct measurements of adsorption alone. This approach captures the overall concentration response associated with biochar contact but does not independently separate adsorption, desorption, and other processes that may affect nutrient concentrations.

Several limitations should be considered when interpreting these results. First, the biochar produced in three pyrolysis reactors was combined into a single bulk stock before the nutrient experiments. Therefore, the three reactors did not constitute independent biochar treatment replicates, and the results do not quantify variability among independently produced biochar batches. Second, the experiments were conducted under controlled laboratory conditions using prepared nutrient solutions. Actual aquaculture water contains dissolved ions, organic matter, suspended particles, and biological communities that may alter nutrient interactions and pH responses. Third, although physicochemical characterization and the pH-drift experiment provided information about the material and its interaction with solutions, the experiments did not directly quantify the contribution of specific mineral phases to alkalinity supply or establish the acid-neutralizing and pH-buffering capacities of the biochar. The source and chemical forms of the released phosphate were also not independently established. In addition, the study did not directly evaluate the effects of biochar on shrimp or fish performance, pond productivity, or long-term water-quality stability.

Conclusions. This study demonstrated that rice husk biochar exhibited nutrient-specific net retention and release behavior under the tested laboratory conditions. Its physicochemical characteristics provided a basis for interpreting these responses, but did not independently establish the mechanisms responsible for nutrient interactions. TAN

showed positive apparent net retention during the first two contact cycles but negative net retention during the third cycle at the selected comparison time, indicating that its performance was not consistent during reuse. Nitrate also exhibited fluctuating retention and readsorption responses, despite positive apparent net retention at the selected comparison time in both phases. In contrast, phosphate showed limited and inconsistent net retention, while its detection in the leaching and desorption experiments indicated that the biochar could also act as a source of dissolved phosphate. Its available phosphorus content may have contributed to this release, although the contribution of the internal phosphorus pool was not quantified independently.

Overall, rice husk biochar should not be considered a universal nutrient adsorbent. Its effects on dissolved nutrient concentrations reflect a balance between retention and release that varies with nutrient type and contact cycle. The biochar also showed potential to influence solution pH, as indicated by its alkaline pH and the pH-drift results; however, its alkalinity contribution and buffering capacity require further quantitative evaluation. These findings provide a basis for investigating rice husk biochar in aquaculture water-quality management, but validation in actual pond water and under operational conditions is necessary before practical recommendations can be made.

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