

Ammonia Reduction by Eco-Friendly Adsorbents Derived from Agro-Industrial By-Products for Shrimp Cultivation

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Abstract

Introduction: Ammonia, a harmful chemical compound, may cause luminescent shrimp and high mortality by triggering shrimp disease, especially from the bacterium *Vibrio harveyi*. This study investigated three different raw materials as adsorbents produced by fast pyrolysis to reduce ammonia levels in shrimp ponds by adsorption.

Methods: Corncoobs, empty palm bunches (EPB), and coconut shells were carbonized by fast pyrolysis at 350, 450, and 550 °C. Characterization analyses included surface area and porosity (BET), morphology (SEM), and functional groups (FTIR). Batch adsorption experiments were conducted under varying pH conditions (pH 6–9) and at different adsorbent dosages (0.4, 0.6, and 0.8 g). Adsorption behavior was evaluated using the Langmuir and the Freundlich isotherm models, as well as kinetic models to determine the adsorption mechanism.

Results: It was revealed that ammonia adsorption was favorable at basic pH. The adsorption uptake was found to increase with the temperature of pyrolysis and contact time, but decrease with the adsorbent dosages. The pseudo-second-order model exhibited the best fit for the kinetic studies, indicating that the chemisorption process limits ammonia adsorption. The Langmuir model best describes the uptake of ammonia, implying that the adsorption of ammonia onto EPBs-char in this study is homogeneous in a monolayer.

Conclusion: All three activated carbons effectively reduced ammonia levels and pH. The alternative activation solution needs to be considered in the future; strong acids will actually add to new waste. Activated-char modifications also need to be developed, taking into account environmentally friendly materials.

Keywords: Toxic compounds, Adsorption, Pyrolysis, Shrimp-Ponds, Wastewater

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Introduction

Ammonia is a hazardous pollutant that poses significant environmental and biological risks in aquatic ecology (1). It originates primarily from the decomposition of organic matter, excess feed, and the excretion of marine animals such as shrimp (2). Elevated ammonia concentrations not only degrade water quality but also disrupt microbial balance, reduce dissolved oxygen, increase water acidity, and promote the proliferation of pathogenic bacteria, notably *Vibrio harveyi* (3).

The bioluminescence phenomenon – commonly referred to as “glowing shrimp” – is linked to luminescent bacteria that emit greenish-blue light via enzymatic reactions involving luciferase and luciferin, as seen in *Vibrio harveyi*. This indicates that the ponds are rich in dissolved ammonia (4,5). This biochemical reaction, controlled by the bacterial lux operon, produces oxyluciferin and light as by-products in the shrimp (6). Although visually striking, the presence of bioluminescent shrimp signals severe ecological

stress (7). This condition leads to disease outbreaks and mass mortality (5) through an impact on aquaculture productivity and farmers’ income (6). Chronic exposure to ammonia—whether through aquaculture wastewater or occupational contact—can lead to respiratory issues, skin irritation, and eye damage in humans (8). According to the U.S. Environmental Protection Agency (EPA), the chronic exposure limit for ammonia in freshwater is 0.017 mg/L to protect aquatic life (9). The World Health Organization (WHO) and the Food and Agriculture Organization (FAO) also recognize ammonia as a priority contaminant in water, with threshold values ranging from 0.2 to 0.5 mg/L for aquaculture systems (10). To mitigate ammonia pollution in aquaculture systems, adsorption has emerged as a promising, low-cost, and environmentally friendly technique (11). Among various adsorption materials, activated carbon has garnered attention due to its high porosity, surface area, and diversity of functional groups (12,13). The effectiveness of activated carbon is primarily



determined by the characteristics of the raw materials and the pyrolysis process used to produce it (14). Smaller pores and greater surface areas enhance the adsorption performance by increasing the interaction between the adsorbent and the target contaminant (15).

Numerous investigations have explored various types of adsorbents for ammonia removal, including fly ash-activated carbon (16), zeolite (17), rice husk-activated carbon (18), sawdust (19), coffee grounds (20), and bentonite (21). However, many of these materials are either costly, location-specific, or not sustainably available in large quantities (22). Thus, the development of abundant, renewable, and efficient bio-based adsorbents remains an ongoing challenge (23,24).

In this study, three locally available agricultural by-products – corncobs, coconut shells, and empty palm oil fruit bunches (EPB) – were selected as raw materials for activated carbon production. These materials represent diverse compositions in terms of cellulose, hemicellulose, holocellulose, and triglyceride content, which may influence pyrolysis behavior and adsorption characteristics (25–27). The activated carbons were produced through fast pyrolysis under varying temperatures and heating durations to optimize their structural and functional properties (28).

This study aimed to develop and evaluate low-cost, sustainable activated carbon adsorbents derived from corncobs, coconut shells, and EPB for effective ammonia removal in shrimp aquaculture systems. The research emphasizes a comprehensive approach, including: (1) Material characterization (surface area, pore structure, functional groups); (2) Batch adsorption experiments under different pH levels and dosages; (3) Isotherm and kinetics modelling (Langmuir, Freundlich, pseudo-first/second-order); and (4) Evaluation of adsorption performance under realistic aquaculture conditions. The novelty of this study lies in the comparative use of three biomass types with distinct biochemical compositions, produced under controlled pyrolysis conditions, and their combined evaluation for ammonia adsorption in aquaculture settings. Unlike many previous studies limited by scarce raw materials, this work offers a scalable and regionally adaptable solution to ammonia pollution in shrimp farming.

Materials and Methods

Chemicals and instrumentation

The chemicals used in this study included 0.1 M hydrochloric acid (HCl) for activation of activated carbon, distilled water for rinsing and dilution, and natural shrimp pond water sourced from coordinates 7°41'05" S 113°51'10" E in East Java, Indonesia. The pond water had an initial ammonia concentration of 20 mg/L and a pH of 9.88, as confirmed by the Water Quality Laboratory of Perum Jasa Tirta II. The following instruments were used: Shaker for agitation during adsorption experiments (Bi-one Clamp Orbital Shaker Model COS-490 at 72 rpm), pH Pen Meter (Constant Water Quality Tester WT 501) for measuring pH, UV-Vis Spectrophotometer (Mettler Toledo – FDA

21 CFR) for determining ammonia concentrations, BET Analyzer (BET Micromeritics Tristar II plus) for surface area and pore size analysis, FTIR Spectrometer for identifying surface functional groups (Shimadzu UV 12801pc), SEM Scanning Electron Microscope (FEG Quanta 650) for morphological characterization. The adsorbents were characterized to determine: (1) Surface area and pore properties using the BET and BJH Adsorption-Desorption method as mentioned in Elsherif et al. (29), (2) Surface functional groups via Fourier Transform Infrared Spectroscopy (FTIR), (3) Surface morphology using Scanning Electron Microscopy (SEM). These analyses were conducted to correlate physical and chemical properties with adsorption performance.

Adsorbent synthesis

Three agricultural waste materials—corncobs (hybrid), coconut shells (*Cocos nucifera* var. Mapanget), and empty palm oil bunches (EPB)—were collected from agro-industrial regions in Malang and Blitar Regencies, East Java, Indonesia. The materials were chopped into 1–2 cm pieces and sun-dried for 3 days to reduce moisture content to approximately 15–20%. Activated carbon production was conducted via fast pyrolysis at temperatures of 350, 450, and 550°C, with durations of 30, 50, and 70 minutes, respectively. The resulting activated carbon was ground into powder using a mortar and pestle and sieved to obtain a uniform particle size. Activation was performed by soaking the activated carbon in 0.1 M HCl (1:10 w/v ratio of activated carbon to HCl) and stirring at 450 rpm for 2 hours. Afterwards, the activated carbon was thoroughly rinsed with distilled water until a neutral pH (≈ 7) was achieved. Finally, the activated carbon was oven-dried at 105°C for 4 hours, then stored for use in adsorption experiments.

Batch experiments

Batch adsorption experiments were carried out using activated corncob-char, coconut shell-char, and EPB-char. Each adsorbent (0.4, 0.6, and 0.8 g) was added to 200 mL of shrimp pond water (20 mg/L ammonia). The samples were stirred at 72 rpm for 15 minutes, following the procedure described by Yuliasari et al. (30). Before treatment, the adsorbents were filtered out. The remaining solution was analyzed for ammonia concentration using a UV-Vis spectrophotometer and for pH using a digital pH meter, as shown in Figure 1. The ammonia removal efficiency (%) was calculated using the following formula:

$$\% \text{ Removal} = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

Where C_o is the initial ammonia concentration (mg/L) and C_e is the equilibrium (final) ammonia concentration (mg/L).

Determine pHpzc

The zero point of charge (pH_{pzc}) is indeed a critical parameter in understanding the surface chemistry

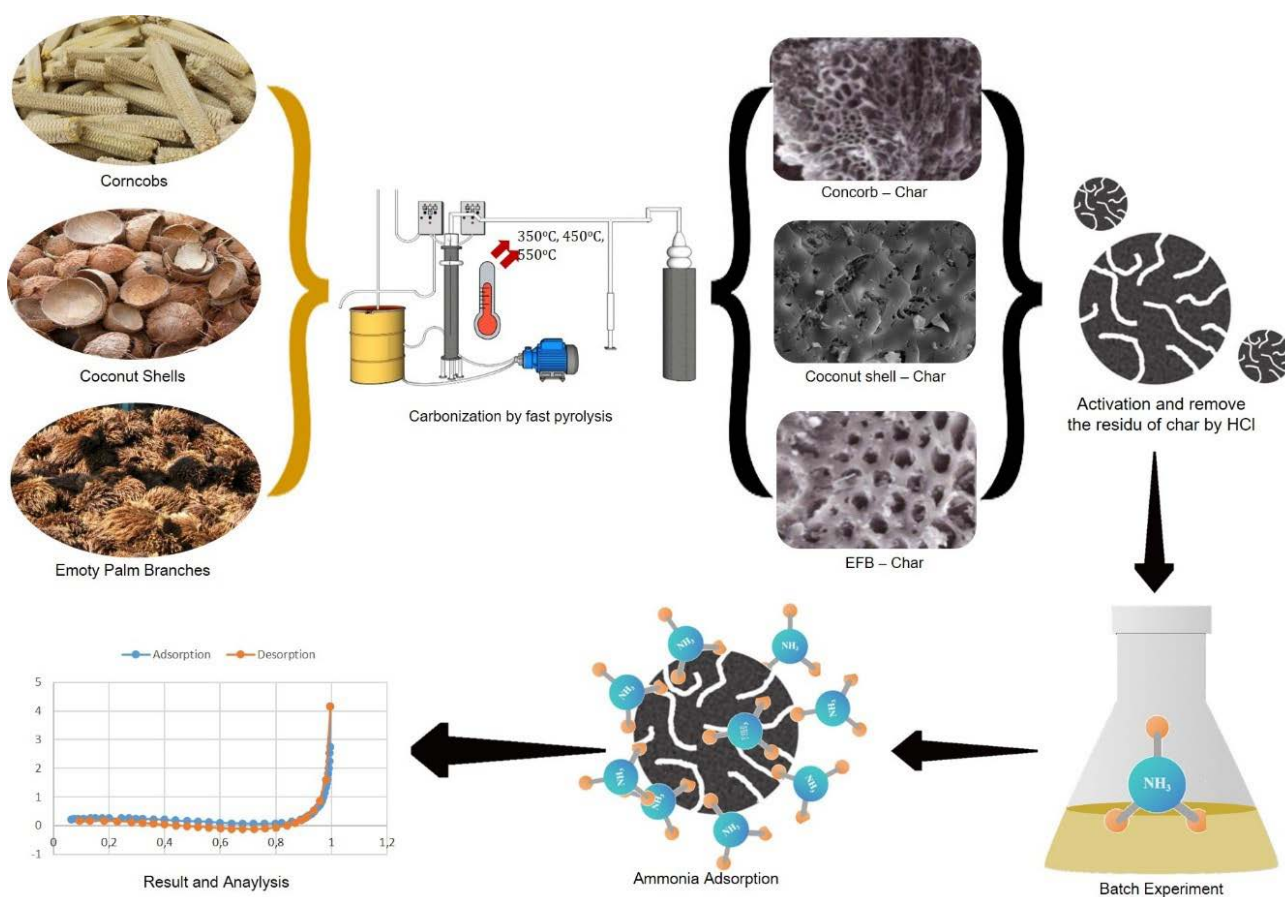


Figure 1. Research workflow schematic

of adsorbents, particularly in relation to electrostatic interactions between the adsorbent and adsorbate. However, in this study, the researcher deliberately focused on evaluating the effects of operational performance due to their immediate practical relevance in real-world applications.

Results

Characterization of adsorbent

Figure 2a, 2b, and 2c show the functionality present in EPB-char, corncobs-char, and coconut shells char before and after ammonia adsorption. Adsorption in the infrared (IR) region occurs at $4,000 - 40 \text{ cm}^{-1}$ due to the rotational and vibrational movements of the molecular groups and chemical bonds within a molecule (31). The FT-IR spectra were obtained to evaluate the chemical structures of three types of biochar qualitatively. Figure 2(a) shows corncobs-char a significant shift in the hydroxyl group absorption band from 2954 to 2026 cm^{-1} , indicating a reduction in hydroxyl group intensity. Similarly, the O-C bond shifted from 1033 to 1038 cm^{-1} , suggesting an increase in carbon-oxygen (oxide) bonding as a result of the adsorption process (32). These changes confirm a transformation in the quantity and nature of hydroxyl groups post-adsorption (33). For coconut shell-char in Figure 2b, a minor shift in the hydroxyl peak from 3049 to 3046 cm^{-1} was observed, along with a change in the O-C bond from 817 to 819 cm^{-1} , indicating similar surface modifications (13). Meanwhile, empty palm bunch (EPB) activated carbon showed in

Figure 2c a more pronounced transformation in hydroxyl groups, with a shift from 3064 cm^{-1} before adsorption to 2612 cm^{-1} after adsorption. These spectral variations across all samples demonstrate that surface functional groups, particularly hydroxyl and oxygen-containing bonds, play a significant role in the adsorption mechanism (34). Figure 2a shows no significant shift in the adsorption peaks; however, the intensity decreases after adsorption. In contrast, Figures 2b and 2c indicate that the intensity increases after adsorption, which is consistent with physical adsorption (35,36).

The Scanning Electron Microscope (SEM) has been a primary tool for characterizing the surface morphology and fundamental physical properties of the adsorbent. As shown in Figures 3a, 3b, and 3c before adsorption, there is a good possibility for ammonia to be trapped and adsorbed into the pores with a size larger than 50 nm , namely, the macropore (37). Macropores in activated carbon facilitate easier diffusion of adsorbate molecules into the adsorbent structure, increase the effective surface area, and accelerate the adsorption process (28). The SEM pictures of the adsorbed samples show distinct dark spots, which can be taken as a sign of effective ammonia adsorption in the cavities and pores of this adsorbent (38). The micrographs presented in 3a, 3b, and 3c, after adsorption, clearly show that the ammonia adsorbed narrows the pore over the entire surface at base pH conditions, resulting in a higher surface area to adsorb other molecules (39).

BET analysis, also known as the Brunauer-Emmett-

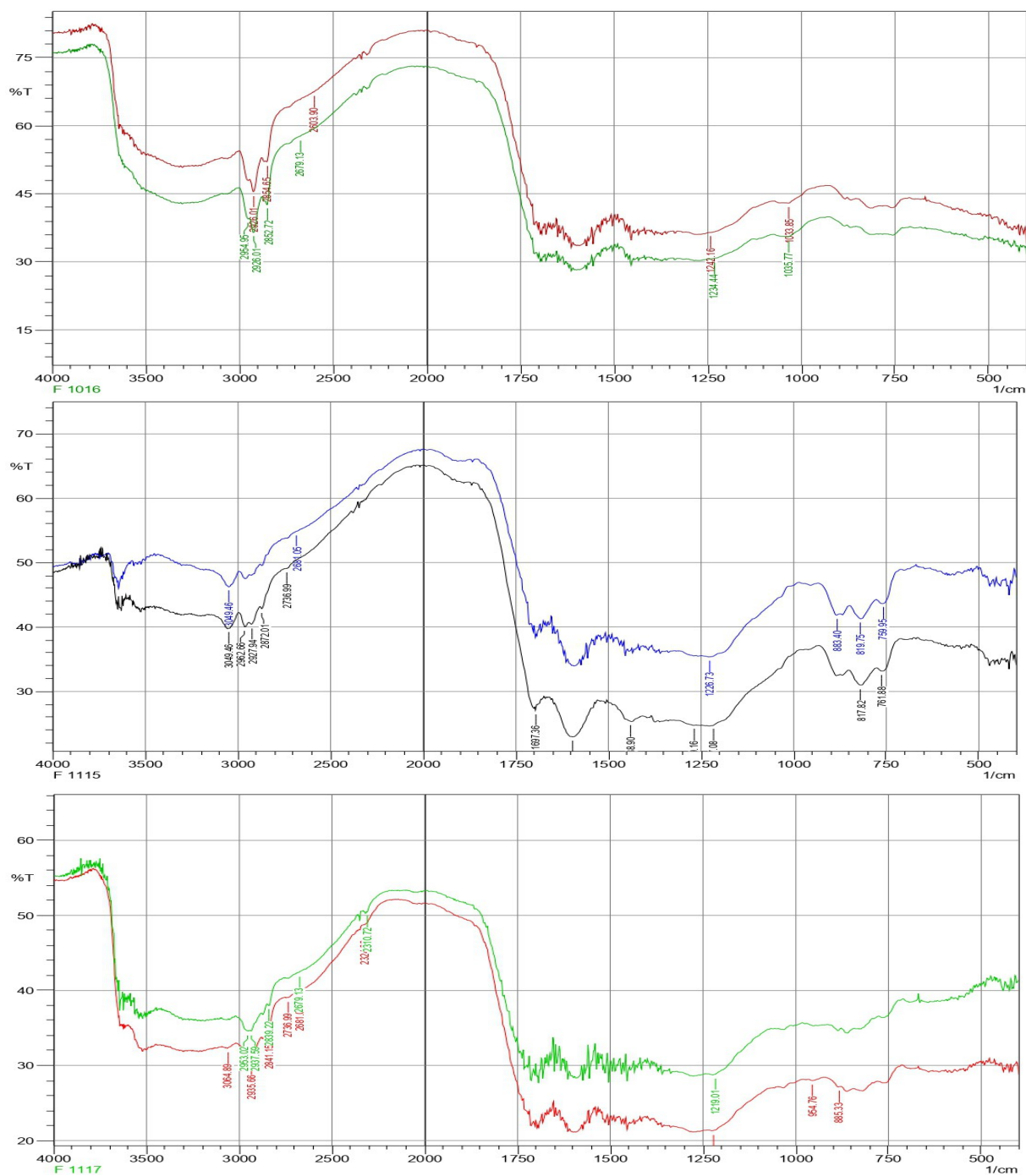


Figure 2. FTIR for adsorbent made from (a) corn cobs, (b) coconut shells, and (c) empty palm fruit bunches for the highest ammonia adsorption capacity.

Teller method, is a scientific technique used to measure the specific surface area and pore size distribution of porous materials. Figure 4 shows that the BET range points for corncobs-char, coconut shells-char, and EPBs-char are in line with $y=0$, indicating that no micropores are formed and the pore range is 2-50 nm and more than 50 nm, which are named mesopore and macropore, respectively. Table 1 presents the measurements of three biochars obtained through BJH analysis, illustrating the pore size range. The highest pore size was obtained in corncobs-char (303,594 Å), followed by EPBs-char (155,136 Å) and coconut shells-char (43,877 Å), respectively.

pH

Adsorption with different fast pyrolysis temperatures, heating times, and masses used affects the pH of wastewater, as shown in Table 2. Overall, the application of adsorbents from the three types of raw materials can reduce the pH in the range of 8.65–9.74 for activated carbon from corn cobs, 9.06–9.87 for activated carbon from coconut shells, and 8.67–9.92 for activated carbon from empty oil palm bunches. Meanwhile, a suitable pH range for shrimp cultivation is between 6 and 9.

Adsorbent dosage

Tables 2 and 3 show the three types of biochars for ammonia adsorption, comparing the masses used to produce the highest adsorption capacity, which were 0.8, 0.6, and

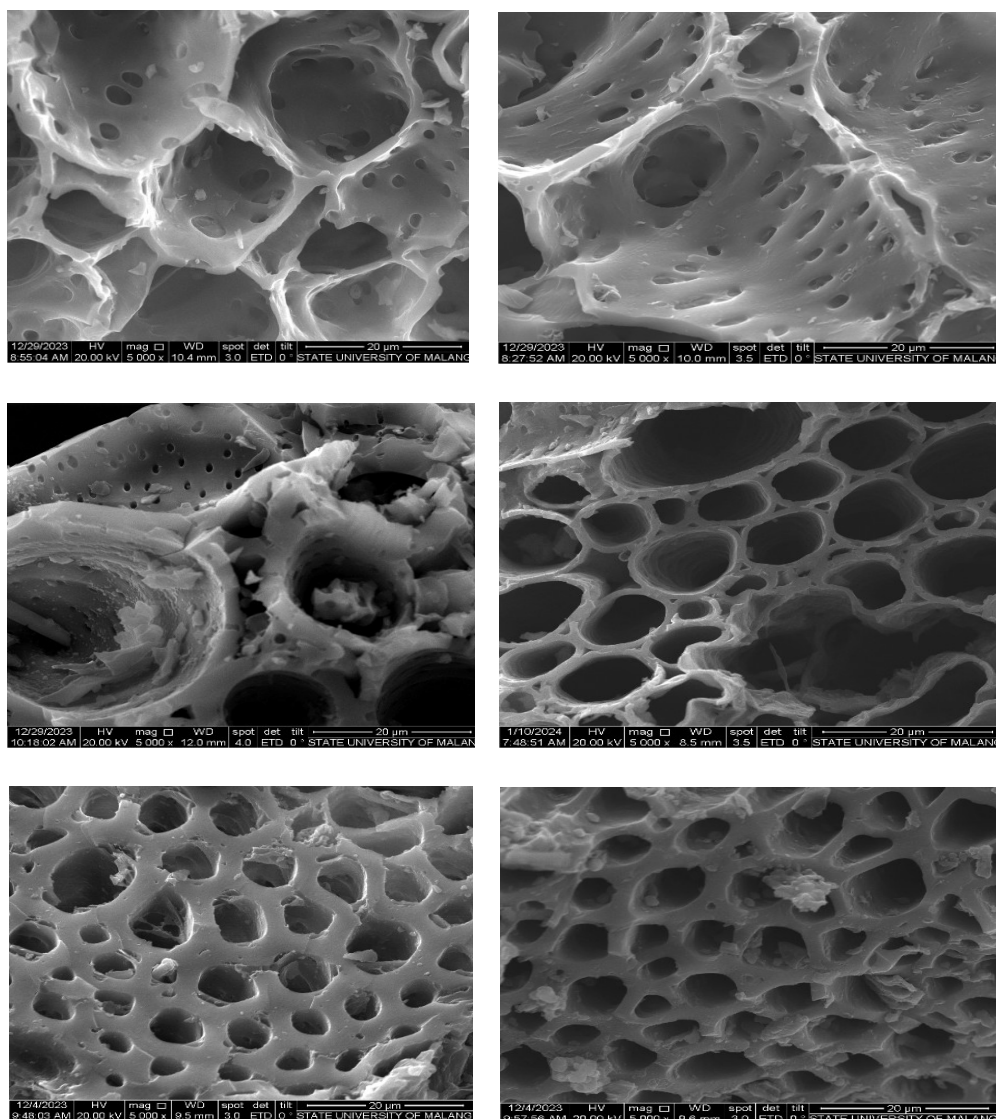


Figure 3. SEM images for (a) corncobs-char, (b) coconut shells-char, and (c) empty oil palm bunches/EPB-char before and after ammonia (NH_3) adsorption

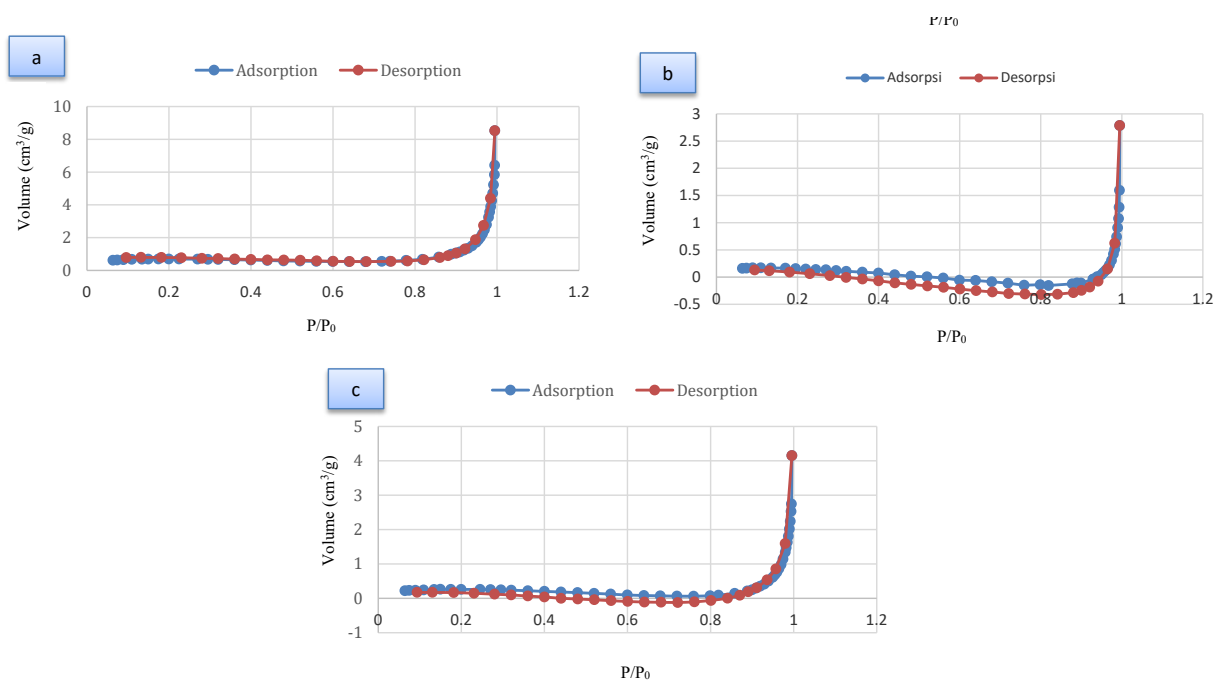


Figure 4. BJH analysis of ammonia adsorption capability and desorption on each activated carbon (a) corncobs-char, (b) coconut shells-char, and (c) EPB-char

Table 1. Specific areas of the three types of biochar.

Parameter	Corn-Char	Coconut Shell-Char	EPB-Char
Single Point Surface area (P/Po)	2.0804 m ² /g	0.6575m ² /g	0.9344 m ² /g
BET surface area	2.0759 m ² /g	0.6724 m ² /g	0.9764 m ² /g
C	-42.429558	597.147319	221.858532
Qm	0.4749 cm ³ /g STP	0.1545 cm ³ /g STP	0.2243 cm ³ /g STP
Pore volume	0.013196 cm ³ /g	0.004084 cm ³ /g	0.004536 cm ³ /g
Pore size			
Adsorption average pore diameter*	254.274 Å	242.922 Å	185.839 Å
Desorption average pore diameter*	57.804 Å	-	46.543 Å
BJH Desorption average pore radius**	303.594 Å	43.877 Å	155.136 Å

Note: (*) 4V/Aby BET, (**) 2V/A.

Table 2. Changes in pH during the ammonia adsorption process using three types of activated carbon at different temperatures, lengths of time, and masses.

Temp (C)	Time (minutes)	Mass (g)	Average of pH Repetition for Corncobs-Char	Std Dev.	Average of pH Repetition for Coconut Shells - Char	Std Dev.	Average of pH Repetition for EPBs - char	Std Dev.
350	30	0	9.64	0.10	9.79	0.07	9.84	0.07
		0.4	9.35	0.28	9.47	0.12	9.31	0.17
		0.6	9.24	0.36	9.43	0.17	9.07	0.18
		0.8	9.13	0.45	9.39	0.16	8.86	0.16
	50	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.32	0.15	9.51	0.18	9.51	0.10
		0.6	9.19	0.20	9.44	0.18	9.39	0.06
		0.8	9.06	0.27	9.36	0.28	9.21	0.03
	70	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.32	0.15	9.50	0.17	9.58	0.06
		0.6	9.20	0.22	9.45	0.18	9.46	0.05
		0.8	9.05	0.36	9.35	0.20	9.38	0.06
450	30	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.41	0.15	9.56	0.15	9.67	0.03
		0.6	9.24	0.24	9.51	0.12	9.64	0.04
		0.8	9.22	0.15	9.48	0.12	9.60	0.03
	50	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.34	0.13	9.57	0.18	9.48	0.03
		0.6	9.30	0.16	9.46	0.17	9.40	0.03
		0.8	9.27	0.16	9.43	0.18	9.19	0.05
	70	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.49	0.11	9.59	0.20	9.51	0.00
		0.6	9.41	0.13	9.54	0.15	9.41	0.03
		0.8	9.28	0.19	9.49	0.12	9.34	0.06
550	30	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.56	0.10	9.50	0.15	9.65	0.11
		0.6	9.54	0.10	9.44	0.13	9.60	0.14
		0.8	9.56	0.15	9.41	0.17	9.51	0.24
	50	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.47	0.23	9.54	0.16	9.54	0.06
		0.6	9.37	0.24	9.49	0.16	9.45	0.08
		0.8	9.32	0.31	9.47	0.14	9.17	0.31
	70	0	9.64	0.10	9.35	0.28	9.35	0.28
		0.4	9.37	0.12	9.44	0.18	9.43	0.03
		0.6	9.30	0.12	9.42	0.17	9.26	0.03
		0.8	9.14	0.34	9.39	0.16	9.12	0.06

Table 3. Ammonia adsorption capacity of three types of activated carbon at different temperatures, lengths of time, and masses.

Temp (C)	Time (minutes)	Mass (g)	Corncobs - char		Coconut shells - char		EPBs - char	
			Average of % Ads	Std. Dev	Average of % Ads	Std. Dev	Average of % Ads	Std. Dev
350	30	0.4	55.72	0.48	59.41	3.11	60.83	0.83
		0.6	64.78	4.62	57.72	4.68	62.30	0.83
		0.8	60.31	7.39	63.61	7.04	62.20	2.06
	50	0.4	56.70	4.10	68.63	9.38	62.65	2.76
		0.6	55.83	3.51	72.95	11.56	65.28	1.69
		0.8	60.34	3.17	63.51	8.86	60.86	1.82
	70	0.4	58.86	2.86	72.18	9.17	64.22	0.57
		0.6	57.57	1.51	60.88	6.47	65.15	1.50
		0.8	64.9	3.59	64.98	5.90	64.55	0.47
450	30	0.4	57.25	3.18	70.59	1.62	85.65	0.97
		0.6	53.40	2.30	65.18	3.47	86.65	1.12
		0.8	52.74	1.96	69.37	1.97	88.20	0.23
	50	0.4	51.74	2.14	64.99	1.37	67.39	4.83
		0.6	56.38	1.16	64.41	1.12	66.57	1.44
		0.8	58.66	3.15	63.55	4.54	64.98	0.50
	70	0.4	59.23	1.02	70.22	1.21	71.09	3.21
		0.6	59.48	1.65	60.06	4.73	62.37	2.72
		0.8	66.57	3.09	64.10	6.62	65.38	1.09
550	30	0.4	61.03	2.19	57.98	9.77	61.03	2.19
		0.6	59.72	0.66	65.74	3.94	59.72	0.66
		0.8	62.53	1.81	65.50	4.61	62.53	1.81
	50	0.4	75.23	0.93	55.61	9.60	75.23	0.93
		0.6	80.38	0.80	61.88	8.25	80.38	0.80
		0.8	81.32	0.53	65.64	1.05	81.32	0.53
	70	0.4	69.95	0.54	60.94	5.67	69.95	0.54
		0.6	71.31	0.50	57.87	6.13	71.31	0.50
		0.8	74.63	0.20	63.24	10.10	74.63	0.20

0.8 g, respectively. To assess the potency of adsorbents in removing ammonia from pond solutions, adsorption fitness was evaluated using respective isotherm models, including Langmuir, Freundlich, and BET, as explained by Elsherif et al. (40).

Ammonia concentration

The percentage removal capability, or adsorption capacity, of the applied activated carbon is shown in Table 3. The initial concentration of ammonia in shrimp cultivation pond water, which is 20 mg/L, can be reduced by adsorbents such as corn cobs, coconut shells, and empty oil palm bunches (EPB), respectively, by 81.32%, 72.95%, and 88.2%. The highest adsorption capacity was obtained from different fast pyrolysis temperatures and heating times, namely at successive temperatures of 450°C for 70 minutes, 350°C for 50 minutes, and 450°C for 30 minutes.

Real sample of shrimp pond

Situbondo Regency has significant potential for cultivating vaname shrimp (*Litopenaeus vannamei*) due to its favorable coastal and environmental conditions. Among the existing aquaculture sites, CV Raja Benur has been identified as

a notable case, particularly due to its relatively high concentration of ammonia, which reaches up to 20 mg/L. The maximum allowable concentration of ammonia in wastewater is 5 mg/L (Minister of Environment Regulation of Indonesia, No. 06 of 2007). As shown in Table 4, the volume of sewage generated during the discharge period consistently exceeds the maximum allowable discharge rate ($D_A > D_M$). This discrepancy indicates a potential risk of ammonia accumulation in the surrounding environment, which could have detrimental ecological impacts (41). To ensure the sustainability and long-term viability of vannamei aquaculture in the region (42), it is essential to implement effective wastewater treatment strategies that can mitigate ammonia pollution and support continuous production cycles.

Kinetics study

Study of adsorption kinetics is essential because the rate of adsorption (which is one of the criteria for efficiency of adsorbent) and also the mechanism of adsorption can be concluded from kinetic studies (43). Table 3 shows that the three types of biochar have varying ammonia adsorption capacity (q). The equilibrium (q_e) was found

Table 4. Conditions of white shrimp ponds.

Parameter	Data
D_p (wastewater flow rate in the shrimp pond)	6.705 m ³ /s (3 ponds are operating, @2.235 m ³ /s each)
h (number of discharge days during the cultivation period)	10 days (during the 70-day production period, siphoning was done 10 times)
D_A (actual liquid waste discharge from the combined production units of two ponds)	67.05 m ³ /s (formula = $D_p \times h$)
q (quantity of wastewater applicable to the type of industry (m ³ /ton) according to the quality standard of Minister of Environment Regulation No. 06 of 2007, m ³ /ton)	30 m ³ /ton
p (amount of raw material used, in this case, stocking density multiplied by final harvest weight. Assumed all survive for the ponds in operation, tons)	340,000 gram – 0.34 ton (formula = 27,200 fry x @12.5 gram)
D_M (maximum wastewater discharge of the combined ponds)	10.2 m ³ /s (formula = $q \times p$)
Conclusion	$D_A > D_M$

in corncob-char, followed by EPB-char and coconut shell-char. To analyze the adsorption kinetics of three types of biochar, the pseudo-first-order (44) and pseudo-second-order (45) models were tested. The results of the fitting are listed in Table 3. A simple kinetic analysis of the adsorption (pseudo-first-order) equation is in the form:

$$q_t = q_e \left[1 - \exp(-k_f t) \right] \quad (2)$$

Where q_t is the amount of adsorbate adsorbed at time t (mg.g⁻¹), q_e is the adsorption capacity at equilibrium (mg.g⁻¹), k_f is the pseudo-first-order rate constant (min⁻¹), and t is the contact time (min).

A pseudo-second-order equation based on the adsorption equilibrium capacity may be expressed in the form:

$$q_t = \frac{k_s q_e^2 t}{1 + k_s q_e t} \quad (3)$$

Where K_s is the pseudo-second-order rate constant (mg.g⁻¹.min⁻¹), the initial sorption rate (h_0 , expressed in mg.g⁻¹.min⁻¹) can be obtained when t approaches zero, in the form:

$$h_0 = k_s q_e^2 \quad (4)$$

Isotherm study

The adsorption isotherm describes the distribution of ammonia between the solid phase and the solution at a given temperature when equilibrium is reached. The Langmuir and the Freundlich models were applied to fit the equilibrium data. Each isotherm model was expressed by certain relative constants that characterized the surface properties and indicated the adsorption capacity of the three materials (46). The Langmuir model proposes that monolayer sorption occurs on the solid surface with identical homogeneous sites (47). It also suggests that no further adsorption occurs once the active sites are covered with ammonia molecules. The following equation presents the saturated monolayer isotherm:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (5)$$

where C_e is the concentration of dye at equilibrium in solution (mg/L); q_e is the unit equilibrium adsorption

capacity; q_m is the maximum dye uptake, giving the information about adsorption capacity for a complete monolayer (mg/g); and K_L is a constant denoting the energy of adsorption and affinity of the binding sites (L/mg).

The Freundlich isotherm is an empirical assumption that the distribution of the heat on the adsorbent surface is nonuniform, namely, a heterogeneous adsorption (48). The equation is stated as follows:

$$q_e = K_f C_e^{1/n} \quad (6)$$

Where n and K_f [mg/g (L/mg)ⁿ] are both the Freundlich constants indicating adsorption intensity and capacity, respectively, the degree of non-linearity between solution concentration and adsorption is n dependent as follows: if the value of n is equal to unity, the adsorption is linear; if the value is below to unity, this implies that adsorption process is chemical; if the value is above unity adsorption, the adsorption is a favorable physical process (40).

Discussion

Structural characterization

FTIR analysis confirmed the presence of hydroxyl (–OH) and C–N functional groups on the activated carbon surface. The interaction with ammonia was evident by changes in transmittance in the 1180–1360 cm⁻¹ range (C–N stretch) and 3300–3500 cm⁻¹ range (–OH). A decrease in intensity near 1638 cm⁻¹ suggests that ammonia attachment occurs via hydrogen bonding and bicarbonate formation, which is a common feature in physisorption mechanisms (31,49). The presence of hydroxyl groups, carbonyl groups, ethers, and aromatic compounds is evidence of the lignocellulosic structure of coconut shell, as also observed in other materials such as Brazilian coconut shell (50) and jackfruit peel waste (51). These findings are consistent with the findings of previous studies that have identified similar binding interactions and peak shifts in ammonia-loaded activated carbon (2, 49). The data suggest that hydroxyl groups play a crucial role in capturing ammonia, likely through the formation of bicarbonate. In all three types of adsorbents, a change in the FTIR spectrum intensity was observed after the ammonia adsorption process (52), attributed to the interaction between ammonia molecules and the active functional groups through hydrogen

bonding, electrostatic interactions, or the formation of coordination bonds. Such interactions can alter the vibrational environment of functional groups, commonly evidenced by changes in peak intensity, peak shifts, or the emergence of new peaks in the FTIR spectrum (24).

Based on pore and adsorption characteristics, the carbon structure of palm empty fruit bunch (EPB) exhibited a greater capacity for ammonia adsorption. SEM analysis further revealed that the pore structure of the EPB activated charcoal was more uniform compared to the other two activated carbon materials. This phenomenon indicates that a larger specific surface area and uniform pore distribution provide more accessible active sites for ammonia molecules. Uniform pores facilitate faster intraparticle diffusion and reduce mass transfer resistance, while the presence of suitable surface functional groups (e.g., $-\text{OH}$, $-\text{COOH}$, $-\text{NH}_2$) enhances interactions through hydrogen bonding and electrostatic forces (53,54). Furthermore, the close match between the molecular diameter of ammonia (~ 0.26 nm) and the micropore of the EPB activated carbon strengthens van der Waals interactions, which would improve adsorption efficiency (43). These findings are consistent with previous studies, which emphasize the critical role of pore uniformity and surface area in enhancing gas adsorption performance (22).

Brunauer-Emmett-Teller (BET) in Figure 4 and Barrett-Joyner-Halenda (BJH) in Table 1 showed that all activated carbons in this study had mesopores and macroporous structures (between 2 and 50 nm and more than 50 nm). This is indicated by the absence of an $x=0$ value in both the adsorption and desorption curves for all three optimal adsorbents used in ammonia removal, as shown in Figure 4. The adsorption-desorption isotherms exhibited H4 hysteresis, characteristic of slit-like pores and capillary condensation in mesopores or macropores (38). SEM analysis confirmed the presence of large, irregular pores before and after adsorption, supporting the interpretation that the materials follow a Langmuir isotherm model and endothermic, spontaneous adsorption behavior (55). Despite variations in pore volume, EPB-char exhibited the highest ammonia removal rate (88.2%), indicating that surface chemistry and functional groups may be more critical than surface area alone under certain conditions.

Effective parameters on adsorption

The results indicate that activated carbon from the three types of raw materials can reduce the pH to meet the best fishery quality standards when heating the raw materials, such as corncobs, coconut shells, and empty oil palm bunches, at temperatures of 350, 450, and 550°C. As the pH of the solution decreases, the adsorption of ammonia was found to increase. This was due to the extinction of positive hydrogen ions, which promoted electrostatic repulsion between the negative charges of the ammonia and the pores of the three types of biochar (13,56). At a basic pH, the functional groups of activated carbon become deprotonated, primarily increasing the negative charge on the biochar surface (carboxyl group, $-\text{COOH}$; hydroxyl

group, $-\text{OH}$; carbon-hydrogen bond, C-H) (57). Carrera-Quintana et al. (2) stated that water pH significantly affects the survival of aquatic biota; changes below or above the threshold will render the chemical toxic. Overall, an adsorbent mass of 0.8 g is more effective at lowering pH than doses of 0.4 g and 0.6 g. For instance, when pyrolysis times and temperatures increased, the pH value decreased at the same dose. Overall, corncob char was able to lower the pH more into the threshold content of the media for shrimp pond cultivation.

Meanwhile, coconut shell-char and EPB-char tend to have a reasonably high pH, making them inadvisable as sole adsorbents; however, both adsorbents will reduce pH optimally when combined (22). The results indicate that activated carbon derived from three different raw materials produced only a minor effect on pH changes. As presented in Table 2, activated carbon from corn cob, coconut shell, and oil palm empty fruit bunch generally showed that the pH after adsorption remained within the range of 9. This finding suggests that a pH range of 7–9 influences the chemical speciation of ammonia, whereby molecular NH_3 is converted into NH_4^+ (40). Ammonia is more readily transformed into positively charged ammonium ions, which can be electrostatically adsorbed onto the negatively charged surface of activated carbon, typically dominated by hydroxyl (OH^-) functional groups (41).

Adsorbent dosage significantly affects adsorption efficiency. The optimum dosages were as follows: Corncob-char, 0.8 g at 450°C for 70 minutes; Coconut shell-char, 0.6 g at 350°C for 50 minutes; and EPB-char, 0.6 g at 450°C for 30 minutes. Higher dosages increase the number of active binding sites, but excessive dosages can lead to site aggregation and reduced accessibility, as reported in previous studies (58, 59). This explains the decreased performance observed at higher dosages, despite a higher available surface area. It is apparent that increasing the adsorbent dose increases the ammonia adsorption capacity, but decreases the amount adsorbed per unit mass. It is readily understood that the number of available adsorption sites increases with an increase in the adsorbent dose, increasing ammonia adsorption capacity (60). The decrease in adsorption capacity per unit mass with an increase in the adsorbent dose is mainly because the adsorption sites remain unsaturated during the adsorption reaction, whereas the number of available adsorption sites increases by increasing the adsorbent dose (29,55).

Although pH_{zpc} (zero point of charge) was not directly measured, pH-dependent trends indicate that activated carbon surfaces carried net negative charges at experimental pH (> 9), facilitating the adsorption of positively charged ammonia species (NH_4^+). Literature supports this mechanism, indicating that pH influences electrostatic interactions and the reactivity of functional groups (61).

Fast pyrolysis was selected due to its efficiency in producing biochar, which features smaller pore sizes that enhance its adsorption potential compared to other thermal conversion methods (48,55,62). Pyrolysis at 350,

450, and 550°C for 30–70 minutes successfully yielded activated carbon with varied physical properties, as shown in Table 3. Notably, a pyrolysis temperature of 450°C with heating durations between 30 and 70 minutes produced the highest ammonia adsorption capacities, ranging from 72.95% to 88.2%. As shown in Table 3, activated carbon derived from corn cobs exhibited optimal adsorption capacity when synthesized through fast pyrolysis at 550°C for 50 minutes with a dose of 0.6 g. This performance was superior to that of activated carbon produced at lower pyrolysis temperatures. This finding is consistent with previous theories suggesting that higher carbonization temperatures reduce pore size and result in a significant increase in surface area (34). This elevated temperature effectively breaks down residual volatile compounds, such as tar and hydrocarbons, which often block the pores of the carbon structure (28).

Furthermore, the application of high temperatures, combined with an activation process, can generate more reactive active sites within the pores (31). For the EPB activated carbon, the highest adsorption capacity was achieved with a dose of 0.6 g at a fast pyrolysis temperature of 450°C for 30 minutes, indicating that this dose was optimal compared to treatments with 0.4 g and 0.8 g. Conversely, for the EPBs-char, no significant increase in adsorption capacity was observed at temperatures of 350 and 550°C. This suggests a saturation point is reached within the activated carbon when the dose is increased. On the other hand, the low adsorption capacity at lower doses indicates the necessity of optimizing the activated carbon dose to match the ammonia concentration in the liquid waste. This aligns with the results of previous research, which found that the active sites of activated carbon have a limited adsorption capacity for compounds that primarily adsorb via physisorption and hydrogen bonding (9). Saturated activated carbon can be regenerated through thermal treatment or washing to restore its active sites (28).

Unlike the activated carbon from corn cobs and EPB, the activated carbon produced from coconut shells, which have a high lignin content (12), showed different optimal conditions. The results indicate that the optimal treatment for coconut shell activated carbon was fast pyrolysis at 350°C for 50 minutes, with a dose of 0.6 g, which performed better than other treatments. However, compared to the other activated carbons produced in this study, the coconut shell-based activated carbon had the lowest overall adsorption capacity. Unlike the activated carbon from corn cobs, the activated carbon produced from coconut shells, which have a high lignin content (12), showed different optimal conditions. The results indicate that the optimal treatment for coconut shell activated carbon was fast pyrolysis at 350°C for 50 minutes, with a dose of 0.6 g, which performed better than other treatments. However, compared to the other activated carbons produced in this study, the coconut shell-based activated carbon had the lowest overall adsorption capacity. This lower performance is likely due to the presence of a significant amount of tar and hydrocarbon compounds blocking the pores, which

restricts the formation of new pores (28). Additionally, the unique and stable nature of lignin makes it particularly difficult to decompose (12), suggesting that a separate activation process with elevated temperatures is necessary to effectively open up the pores and enhance reactivity (31).

Corn-cob-char, with a surface area of 2.0759 m²/g, showed lower adsorption efficiency compared to coconut shell-char and EPB-char, despite having a higher pore volume (0.013196 cm³/g vs. 0.004084 and 0.004536 cm³/g). This may be due to early surface saturation, as indicated by its negative BET isotherm constant ($C = -42.43$), suggesting low affinity for continued adsorption (44). Conversely, the positive constants for coconut shell-char ($C = 597.15$) and EPB-char ($C = 221.86$) indicate greater potential for prolonged adsorption activity (46). The observed pH changes after adsorption reflect the reduction of ammonia and are influenced by the type of activated carbon, dosage, and pyrolysis temperature (63). While pH declined slightly, final values remained above 9, which is hazardous in shrimp aquaculture due to oxygen depletion and elevated toxicity levels (64). Higher adsorbent dosages reduced the pH more effectively, but still did not bring the levels into the safe range (pH 7–8).

Shrimp pond characteristics

The aquaculture of vannamei shrimp (*Litopenaeus vannamei*) in Situbondo Regency is strongly supported by favorable coastal geomorphology, saline water resources, and a tropical climate that enables year-round culture. Shrimp ponds in this region are predominantly earthen-based, rectangular in shape, and range in size from 0.5 to 2 hectares (1.2 to 4.9 acres) (65). Most are designed with peripheral embankments and rely on pumping systems for water exchange, although some ponds still utilize tidal flows during the dry season. Aeration devices are commonly installed to maintain dissolved oxygen levels, particularly under intensive stocking conditions, which often exceed 100 post-larvae per square meter (5,6). While these structural and operational features support high productivity, they also intensify waste generation and nutrient loading within the pond system.

Empirical evidence from CV Raja Benur illustrates the typical challenges associated with intensive shrimp culture in Situbondo. The discharge water has been recorded with ammonia concentrations reaching 20 mg/L, substantially exceeding the regulatory threshold of 5 mg/L as stipulated by the Indonesian Ministry of Environment (Regulation No. 06/2007). In addition, the volume of effluent produced during discharge cycles consistently surpasses the maximum allowable discharge rate ($D_A > D_M$) as shown in Table 1, suggesting that effluent management practices remain inadequate to control pollutant loading (7,11). Elevated ammonia levels are primarily driven by high feed inputs, the accumulation of feed residue and faecal matter, and the microbial decomposition of organic residues. These conditions increase the risk of eutrophication in receiving waters, disrupt ecological balance, and simultaneously impair shrimp health due to ammonia toxicity (18).

When compared with other shrimp-producing regions in Indonesia and Southeast Asia, the pond characteristics in Situbondo share many similarities but also reveal context-specific vulnerabilities (49). For instance, in East Java and Lampung, shrimp ponds often employ semi-intensive systems with larger pond sizes and partial water recirculation, which can reduce effluent discharge volumes (66). In contrast, many farms in Thailand and Vietnam have adopted lined ponds with biofloc or recirculating aquaculture systems (RAS), which significantly improve water quality control and reduce nutrient loading into the environment, as shown in [Figure 3](#). The predominance of earthen ponds in Situbondo, while cost-effective, limits effluent treatment capacity and contributes to higher pollutant release (18). These differences highlight the need for region-specific interventions: whereas advanced systems in Thailand and Vietnam prioritize technological intensification (67), shrimp farming in Situbondo may benefit from transitional approaches such as the integration of sedimentation ponds, constructed wetlands, or biofiltration systems before discharge, as shown in [Figure 5](#).

Kinetics studies and isotherm

Kinetic modelling helps define the rate-limiting steps of adsorption, as shown in [Figure S1](#). The pseudo-second-order model fitted the experimental data better than the pseudo-first-order model, with R^2 values of (1) Corncob-char: 0.9987; (2) Coconut shell-char: 0.9993, and (3) EPB-char: 0.9997. Calculated and experimental ($q_{e, \text{exp}}$) values closely matched, indicating that physical interaction (physisorption) was dominant (45). The tendency of the adsorbents to follow a pseudo-second-order model also shows that the active sites on the adsorbent are sufficiently abundant (56), allowing for the adsorption of a greater amount of adsorbate even at relatively low concentrations (48). This supports the potential application of adsorbents for removing not only ammonia but also other contaminants in various types of liquid waste (68,69). [Figure S1](#) represents the variation of ammonia adsorption on three types of biochar with shaking time (0-60 min) and initial ammonia solution concentration of 20.0 mg/L. It is indicated that while the adsorption of ammonia was relatively rapid initially, the rate of adsorption gradually

slowed over time and reached a constant value (equilibrium time) (40,46). The initial faster rate may be due to the availability of the uncovered surface area of the adsorbents (45).

Isotherm modelling revealed that the Langmuir isotherm provided the best fit for all three activated carbons, with R^2 values close to 1. [Table S1](#) shows that the Langmuir and Freundlich adsorption models fit the experimental data for ammonia adsorption sufficiently, as indicated by the linear correlation coefficients (R^2). The most significant values of R^2 (0.999 for corncoobs-char; 0.984 for coconut shells-char; 0.997 for EPBs-char) were obtained in Langmuir models compared with the Freundlich models (0.986 for corncoobs-char, 0.936 for coconut shells-char, 0.992 for EPBs-char), indicating the applicability of the Langmuir isotherm for ammonia adsorption (47). This suggests monolayer adsorption on a homogeneous surface, which means that the adsorbent can become saturated and the adsorbate can be released, allowing for potential reuse.

The Langmuir isotherm also includes a derived formula to determine the separation factor (RL) as shown in [Table S1](#), which indicates the effectiveness of the adsorption process: (1) Corncob-char: 0.347; (2) Coconut shell-char: 0.079, and (3) EPB-char: 0.153. These values ($0 < RL < 1$) confirm favorable and efficient adsorption, in line with previous findings in activated carbon research (25, 30, 41). The different K_L values among corncoobs-char, coconut shells-char, and EPBs-char refer to the differences in binding strength and capacity of the ammonia with the surface of the biochars; in general, K_L decreased with the rise of pressure and temperature (33). The parameters of K_f and $1/n$ exhibited intense change at higher temperatures. The values of $1/n$ ($0.1 < 1/n < 1$) indicated favorable adsorption of three types of biochars under experimental conditions (0.784 for corncoobs-char, 0.591 for coconut shells-char, 0.126 for EPBs-char). By comparing two models, the Langmuir model is a suitable adapted model for the fitting of adsorption isotherms.

The q values calculated ($q_{e, \text{cal}}$) from the pseudo-first-order model were more consistent with the experimental q values ($q_{e, \text{exp}}$) than those calculated from the pseudo-second-order model, as presented in [Table S2](#). Moreover, the experimental data showed better compliance with the

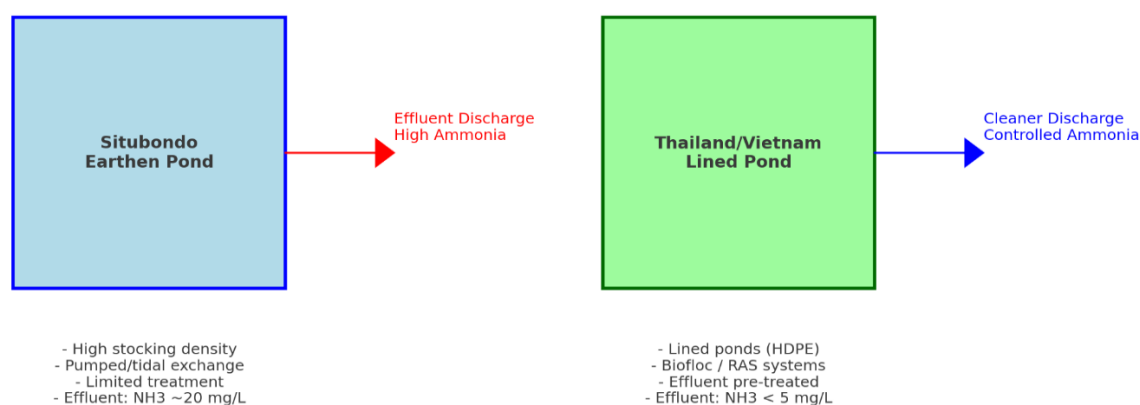


Figure 5. Comparison of Shrimp Pond Characteristics: Situbondo and Thailand/Vietnam, modifications from (67)

chemisorption kinetic model for ammonia adsorption in terms of higher correlation coefficient values R^2 in three types of biochar: 0.98997 for corncobs-char; 0.9993 for coconut shells-char; 0.997 for EPBs-char, respectively.

Comparing with other studies

These findings indicate a higher value for ammonia adsorption than the adsorbents listed in Table 5.

On the other hand, this study achieved a pH value of greater than 9, which is higher than in other studies, but still within the range of pH levels found in wastewater (70).

These results indicate that an alkaline pH enhances NH_4^+ adsorption due to favorable speciation and surface charges. However, there are variations depending on adsorbent modification and system specifics (34). For example, in batch adsorption studies using activated carbon derived from waste types, optimal ammonia nitrogen removal was observed at pH 9.0 (9). Similarly, activated alumina has demonstrated maximum ammonium ion adsorption at a pH of 9.0 (10). For instance, iron-loaded activated carbon exhibited optimal ammonium removal (up to ~91%) at pH 8.11, underscoring the importance of electrostatic interactions under near-neutral conditions (36). In another study employing general biosorption techniques, the highest ammonia removal occurred at around pH 6, owing to enhanced cation exchange, while lower pH values suffered from competition by H^+ ions (37).

As presented in Table 3, the reduction of ammonia concentration through adsorption using three types of activated carbon produced at different fast pyrolysis temperatures demonstrates that biomass-based activated carbon possesses high potential as an adsorbent for hazardous waste. Among the tested materials, activated carbon derived from oil palm empty bunch (EPB) at 450°C exhibited the highest ammonia adsorption capacity, achieving 88.20% removal with a final ammonia concentration of 2.36 mg/L. This value complies with the wastewater quality standard established by the Indonesian Ministry of Environment, as outlined in Regulation No. 6/2007. In comparison, activated carbon derived from corn cobs and coconut shells exhibited adsorption capacities of 81.32% and 72.95%, respectively, resulting in final ammonia concentrations of 3.68 and 5.41 mg/L, respectively. These

concentrations exceed the permissible ammonia discharge limits for aquaculture waters. The superior performance of EPB-based activated carbon is attributed to its tubular mesoporous structure, which enhances the effectiveness of ammonia removal. The maximum adsorption capacity observed in this study surpasses that of EPB activated carbon modified with H_3PO_4 , which achieved 84.53% removal (54).

Furthermore, Nurmasari (55) reported an adsorption capacity of only 70.79% in her study, highlighting the advantage of the mesoporous tubular structure of EPB-based activated carbon. This morphology provides a relatively large specific surface area and extensive surface accessibility, thereby facilitating higher ammonia uptake (56). Additionally, tubular mesopores exhibit significantly faster adsorption rates compared with pore structures dominated by cracks or transverse cavities (31).

Adsorption mechanisms

Both solution chemistry and the properties of the adsorbent surface strongly influence the adsorption mechanism of ammonia onto activated carbon. At pH values between 7 and 9, ammonia predominantly exists in the protonated form as NH_4^+ , which facilitates electrostatic attraction with the negatively charged functional groups on the carbon surface, such as hydroxyl ($-\text{OH}$), carboxyl ($-\text{COOH}$), and other oxygen-containing moieties. This ionic interaction is further enhanced by the presence of mesoporous structures, as observed in the oil palm empty bunch (EPB)-based activated carbon, where tubular mesopores provide greater accessibility and higher surface area for NH_4^+ ions to diffuse and bind. In addition to electrostatic interactions, physical adsorption through van der Waals forces and pore filling contribute to overall ammonia removal, particularly in micropores where molecular sieving can occur. The synergistic effect of surface charge, pore architecture, and functional group distribution explains why EPB-derived activated carbon at 450°C demonstrated superior adsorption performance compared with corn cob and coconut shell carbons. These combined mechanisms indicate that both chemical speciation of ammonia and the structural characteristics of the adsorbent are critical determinants of adsorption efficiency.

Table 5. Comparison of ammonia adsorption

Adsorbent Material	Mass of adsorbent (g)	Effect of the pH	Capacity of Ammonia removal (%)	Ref
Corncoobs-char	0.8	9.32	81.32	This study
Coconut shells-char	0.6	9.43	72.95	This study
EPBs-char	0.8	9.60	88.2	This study
Baggage fly ashes	2.5	7	45.73	(71)
Carbon-plastic ore composite	3	8	43.01	(72)
Zeolit	5	7.5-8	58.44	(73)
Carbon – coffee grounds	3	5	66.57	(74)
Carbon – rice husks	5	7	75.87	(30)
Coal ash- TiO_2 composite	3	7-7.3	79	(8)
Bentonite	10	7-7.5	82.04	(75)

Figure S2 shows that despite its superior adsorption capacity, the application of EPB-derived activated carbon also warrants a critical evaluation regarding operational conditions and long-term stability. The efficiency of ammonia removal is strongly influenced by solution chemistry, particularly pH and ionic strength, which govern the equilibrium between NH_3 and NH_4^+ species. While alkaline conditions (pH 7–9) favor the conversion of free ammonia to ammonium ions and enhance electrostatic adsorption, extreme variations in pH may reduce adsorption efficiency due to competition with protons (H^+) or other cations present in aquaculture wastewater (37). Additionally, repeated adsorption–desorption cycles may lead to pore blockage, surface fouling, and a reduction in the activity of functional groups, ultimately diminishing the adsorbent's reusability (76). Previous studies on biomass-derived adsorbents have also emphasized the importance of regeneration performance, as adsorption efficiency often declines by 15–30% after multiple cycles without proper treatment (57). Furthermore, large-scale implementation requires consideration of production costs, energy consumption during pyrolysis, and the environmental footprint of adsorbent preparation. In this regard, integrating EPB-based activated carbon with complementary treatment strategies, such as biofiltration or advanced oxidation, may provide a more robust and sustainable solution for ammonia management in aquaculture systems.

Conclusion

Activated carbon derived from corncobs, coconut shells, and empty palm oil fruit bunches (EPB) is a sustainable and cost-effective adsorbent for removing ammonia from shrimp pond wastewater. The shifting of peaks in the FTIR spectrum confirmed the adsorption of ammonia onto three biochars. The SEM study also provided support for this by observing differences in the surface morphology of the adsorbent before and after adsorption. Kinetic adsorption models for three biochars adsorbing ammonia were followed by a simulation of the adsorption study, indicating that chemisorption followed pseudo-second-order kinetics. The equilibrium data were best fitted to the Langmuir model. The adsorption process was controlled by monolayer mechanisms, involving a chemisorption process and homogeneity. Future research should further enhance the adsorption performance through physical or chemical activation, optimization of pyrolysis conditions, and integration with complementary treatment methods such as biological filtration.

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Competing Interests

The authors affirm that they have no known financial conflicts of interest or personal relationships that could have affected the results presented in this journal.

Ethical Approval

This study complies with ethical guidelines and principles, prioritizing the protection of human subjects, proper management of hazardous materials, and maintaining scientific integrity. Informed consent was obtained from participants, conflicts of interest were transparently disclosed, and participant privacy and confidentiality were safeguarded. The authors are dedicated to upholding ethical standards in both research and reporting, contributing responsibly to the advancement of knowledge.

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Supplementary Files

Supplementary File 1 contains Tables S1–S2 and Figures S1 and S2.

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