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Nitrate Adsorption Using Activated Carbon from Agricultural Waste

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ABSTRACT

Nitrate (NO_3^-) *contamination poses a significant threat to aquatic ecosystems, as it contributes to eutrophication, decreases water quality, and poses risks to human health. Synthesis of activated carbon from agricultural waste provides a circular solution for wastewater treatment and nitrate removal. Adsorption is an effective and simple method for reducing nitrate concentration in water. This study investigated nitrate removal in a batch experiment using a mixture of corn cob and coconut shell using KOH activation. A one-factor-at-a-time (OFAT) approach was used to study the influence of adsorbent dose (30-70 mg) and adsorption contact time (10-60 minutes). The activated carbon was characterized by Fourier transform infrared spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). The nitrate removal efficiency was analyzed using a UV–Vis spectrophotometer at 540 nm. The characterization results showed that the activation process increases the specific surface area, pore volume, and number of active sites for nitrate adsorption. Under optimized conditions, nitrate removal efficiency reached 99.37%. This study confirms the effectiveness of agricultural waste-derived activated carbon as a sustainable adsorbent for nitrate adsorption in water treatment applications.*

INTRODUCTION

Water contamination has become a critical environmental problem, especially in agricultural and industrial areas. Nitrate is a major component of agricultural fertilizers. Excess nitrate levels cause serious health and ecological problems, such as methemoglobinemia, cancer, massive alga growth, and eutrophication. The Environmental Protection Agency (EPA) has established a maximum level of 10 mg/l NO_3^- in drinking water, which is more stringent than the World Health Organization's (WHO) recommendation of 50 mg/l NO_3^-. Consequently, the removal of nitrate is a significant issue (Mahmoud et al., 2024).

Commonly used technologies in the remediation of nitrates include ion exchange, electrodialysis, biological denitrification, as well as various adsorption methods. However, the high cost of this technology makes its use limited in the treatment of nitrates in water. In addition, denitrification takes a long time as well as a large area to achieve optimal results (Chang et al., 2023). Among the various nitrate remediation methods, the adsorption technique is an effective solution to remove nitrate pollutants in water treatment. Adsorption is a simple technique, with high removal efficiency, low operational costs, and lower waste (Moon et al., 2025).

Through adsorption techniques, agricultural waste is considered a practical, affordable, and sustainable resource in large-scale biochar production. Biochar is a carbon-rich material produced through the pyrolysis process of organic matter under limited oxygen conditions. Biochar has physicochemical properties, including specific surface area, functional group content, and porosity, that play a role in determining its effectiveness in various applications. The use of agricultural waste as raw materials is considered efficient as a sustainable solution to convert residual biomass into high-value products. (Sama et al., 2025).

Among the most common agricultural wastes, coconut shells and corn cobs are among the most abundant. Globally, corn is the main cereal crop with a total production of 594 million tons from an area of 139 million ha. Post-harvest, it is estimated that 8.6-8.9 Mg/ha of biomass is left behind in the field. Corn cobs contain about 28.7–41.3% cellulose, 39.3–46.0% hemicellulose, and 7.4–19.6% lignin. (Bélanger et al., 2023). Coconut shell activated carbon also has promising capabilities in water treatment. Previous research has shown that coconut shell activated carbon can remove heavy metal ion pollutants by more than 98% and is effective in removing amoxicillin, ciprofloxacin, and carbamazepine by up to 70% (Saad et al., 2024). Coconut shells have developed pores with strong adsorption properties, higher resistance, easy regeneration, and potential in wastewater remediation by the manufacture of activated carbon (Chen et al., 2024). Therefore, the combination of these materials offers a high lignocellulosic content of corn cobs and a porous structure of coconut shell, which collectively provide a balanced carbon matrix. This makes it a promising candidate for sustainable activated carbon resources

The adsorption capacity can be enhanced through the activation process during pyrolysis. The activation process serves to remove impurities and open the pores of activated carbon (Yuningsih et al., 2024). Modification of raw materials needs to be done to change the properties of the surface functional group. Modifications are tailored to the targeted contaminants so that the resulting product is in accordance with the desired application (Liu et al., 2022). Chemical or physical modification has proven to be a good adsorbent. Chemical activation shows advantages over physical activation, such as shorter time, higher surface area, and higher product. To perform chemical activation, biomass needs to be impregnated with an activating agent, such as NaOH, KOH, H_3PO_4, or $ZnCl_2$, before being carbonized. KOH is often the top choice due to its environmentally friendly synthesis process and brings high surface area and varied functional group content, making it an effective candidate wastewater treatment applications. (Jawad et al., 2021). The contributions of corncob and coconut shell characteristics can be optimized by mixing the precursors at a 1:1 ratio, thereby maximizing pore-structure and enhancing thermal stability during the pyrolysis.

This research focuses on optimizing nitrate ion adsorption using activated carbon from corn cob and coconut shell mixture adsorbents using a one-factor-at-a-time (OFAT) method. The resulting activated carbon will be characterized using FTIR, SEM-EDX, and XRD to identify the carbon surface. The adsorption parameters described are contact time, adsorbent dosage, and initial sample solution concentration. The adsorption results are measured using a UV-Vis spectrometer to determine the effectiveness of nitrate removal in water.

RESEARCH METHOD

Chemicals

Deionized Water (DW), Potassium Hydroxide Solids (KOH) 65%, Chloride Acid (HCl) 37%, Potassium Nitrate Powder (KNO_3), Zinc Powder, Sulfanilamide, N-(1-

Naphthyl)-ethylenediamine dihydrochloride/NED, Glacial Acetic acid (CH_3COOH), Sodium Acetate Trihydrate ($CH_3COONa \cdot 3H_2O$) manufactured by Merck. Briefly, a nitrate stock standard solution (1000 mg L^{-1}) was prepared by dissolving 0.1631 g anhydrous potassium nitrate (KNO_3) in 100 mL of deionized water.

Griess reagent was prepared from sulfanilamide and NED solution. 1 gram of sulfanilamide was dissolved in a mixture of 10 ml of concentrated HCl with 50 ml of deionized water. Then add aqua dm up to 100 ml. 0.1 grams of N-(1-Naphthyl)-ethylenediamine dihydrochloride/NED is dissolved in 100 ml of deionized water, and this solution will be added to the analyte to synthesize the nitrite so that it becomes purplish-red (Jiwarungrueangkul et al., 2023). A 0.75 mol L^{-1} acetate–acetic acid buffer at pH = 6.10 was prepared by dissolving sodium acetate and glacial acetic acid in water, and the pH was set to 6.10 using 10 mol L^{-1} sodium hydroxide (da Ascenção et al., 2024).

Preparation of Adsorbents

In this work, the corn cob (CC) and coconut shell (CS) were applied as a precursor for biochar production. The corn cob and coconut shells were washed using deionized Water to remove the impurities. Then dried under sunlight before thermal drying at 110 °C for 24 h. The adsorbent is smoothed with a grinder mill using a disk mill strainer type FFC size 1.5 and then 0.3 mm, and then sifted with a 250 μm sieve.

Then, CC and CS are mixed in a ratio (1:1), then activated with KOH in a ratio of 1:4. The Corn Cob and Coconut Shell (CCCS) mixture is left for 24 h and dried at 80 °C for 24 h. The mixture was continuously transferred into a crucible for pyrolysis at 600 °C under nitrogen conditions for 2 h to achieve activated carbon. After activation, the carbon was rinsed with demineralized water and drying at 80 °C for 24 hours to ensure impurity removal.

Characteristics of Biochar

The surface morphology characteristics and the elements constituting of adsorbent were analyzed by scanning electron microscope (SEM) (S-4800, Hitachi, Japan). The functional group was analyzed using Attenuated Total Reflectance-Fourier Transform Infrared spectroscopy (ATR-FTIR; Agilent).

Batch Adsorption Studies

This adsorption study aims to investigate the influence of experimental conditions on NO_3^- adsorption and determine the maximum amount of NO_3^- removal. The adsorption procedure was conducted using a batch model by adding an adsorbent with a mass according to the design and 25 mL of nitrate solution with an initial concentration. The mixture is stirred at 200 rpm for a predetermined time (Fang et al., 2023). The parameters measured were adsorbent dose (30-100 mg), initial nitrate concentration (25-100 mg/L), and contact time (30-120 min). The concentration of NO_3^- was determined by the spectrophotometric method using the Griess method at wavelength λ = 540 nm (da Ascenção et al., 2024)

Adsorption optimization is carried out based on the *One-Factor-At-A-Time* method to analyze the effect of each factor on the % removal and adsorption capacity of nitrate ions. The efficiency of NO_3^- was determined by the ratio of the differences between the initial (C_0) and final concentration (C_e) (Eq. (1)).

$$R\% = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

The adsorption capacity qe (mg/g) was determined with Eq. (2) :

$$qe = \frac{C_0 - C_e}{m} \cdot V \quad (2)$$

Where m is the adsorbent dose and V is the volume of solution (L) (Rahdar et al., 2021)

RESULTS AND DISCUSSION

a. Material Characterization

1) SEM analysis

The surface structure of the synthesized coconut shell corn cob activated carbon (CCCS) was analyzed using SEM to analyze the effects of modifications on the carbon surface morphology. Fig. 1 shows how the surface morphology is activated with KOH. SEM results show changes in material morphology during the modification process with KOH. The raw material (Fig. 1.a) shows a solid bulk surface with limited porosity. This shows a low surface area and limited active sites. The disadvantage of agricultural waste in nitrate removal is its relatively low adsorption capacity, for which it is necessary to carry out physical or chemical modifications to increase it (Kalaruban et al., 2016).

Post-activation with KOH (Fig. 1b), irregular protrusions are formed on the surface, indicating that the active side of the carbon has been formed. This transformation is associated with a chemical reaction between KOH and the carbon matrix that promotes the formation of micropores and mesopores, as well as functional groups that increase the porosity of the surface and the active side of carbon. KOH activation enables interaction between the activator and biomass by breaking the bonds of lignocellulose bonds. This process increases porosity and modifies surface area to improve adsorption efficiency (Setiawan et al., 2025).

Post-pyrolysis under an N_2 atmosphere (Fig. 1c) reveals a more stable porous structure, indicating that the carbon undergoes pore formation and strengthening during thermal treatment. During the pyrolysis process, the combined effects of chemical activation, oxidative etching, and pore widening are effective in increasing porosity, increasing surface area, and creating micro and macroporous structures. Post-pyrolysis, it shows a more porous structure with a rough texture, and many macro pores are formed. This is due to the alkaline properties that dissolve ash and mineral substances, which block pore structures of biochar (Premchand et al., 2024).

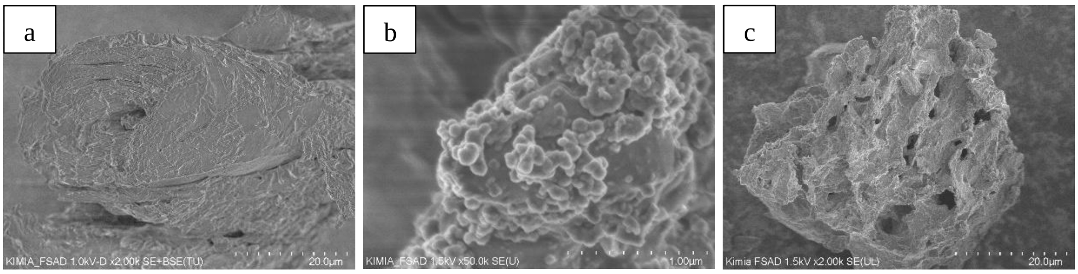

Figure 1. SEM patterns of KOH modification : (a) raw material, (b) after activation with KOH ; (c) after pyrolysis under N_2 conditions.

2) FTIR analysis

Fourier transform infrared spectroscopy (FTIR) analysis can provide information related to the structure and composition of chemical compounds. As shown in the Table. 1, The FTIR spectrum of raw materials shows an O-H band at 3330 cm^{-1}, also showing the presence of hydrogen bonds in the lignocellulose matrix. Furthermore, the bands at 2924 cm^{-1} (C–H) and 1736 cm^{-1} (C-O) represent the characteristic vibrational peaks of cellulose and hemicellulose (Sun & Webley, 2010).

After KOH pyrolysis activation, the O-H band shifted at 3408 cm^{-1}, which indicated that the KOH reaction resulted in the formation of a new hydroxyl group and indicates the process of dehydration. In addition, the appearance of new bands at 1631 cm^{-1} (C-O) and 1374 cm^{-1} (C-H) indicates dehydration of functional groups. The formation of C≡N as active nitrogen groups that appeared at 2107 cm^{-1}. The chemical alteration and breakdown of the surface, as observed through changes in absorbance, are caused by KOH activation (Mariana et al., 2021). The presence of an oxygen-containing group, such as O-H and C-O, enhances electrostatic attraction between nitrate ions. These contribute to surface interaction and increase the adsorption capacity (Eissa et al., 2024).

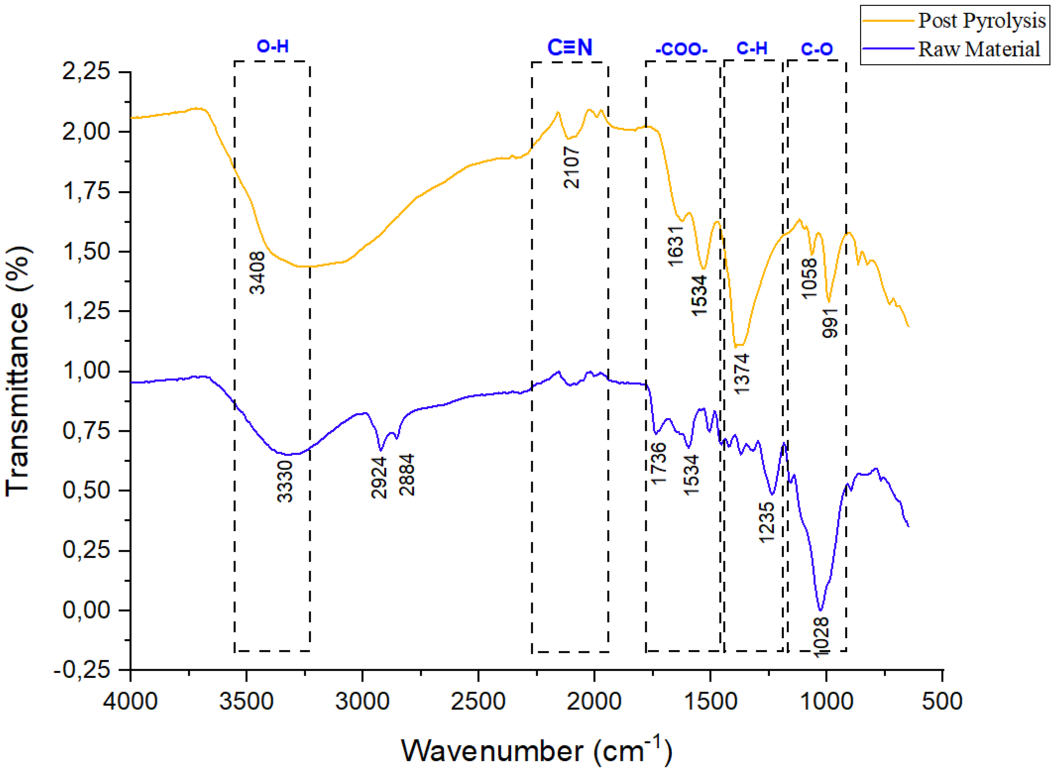

Figure 2. FTIR spectra of raw material (corn cob-coconut shell) and after pyrolysis

b. One-Factor-At-A-Time Result

1) Effect of contact time

The 50 mg/L initial concentration and 30 mg adsorbent dose, in Figure 3, the adsorption efficiency decreases significantly during the first 30 minutes, with the high removal reaching 99,.37% (10 min) with 41.40 mg/g adsorption capacity. This result confirmed that the rapid adsorption of NO_3^-, and the equilibrium was achieved within 30 minutes of contact time, indicating a relatively constant adsorption capacity (qe) value. KOH reacts with carbon to form compounds such as K_2CO_3, K_2O, CO_2, and H_2 that widen the carbon matrix (mesopores and macropores). This surface development accelerates the diffusion of nitrate ions in the internal pores and facilitates transport to the active side so that equilibrium is quickly achieved (Nayak et al., 2017).

These rapid adsorption kinetics show advantages over using a single KOH-activated corn cob-based adsorbent. For comparison, a previous study reported that KOH-activated corn cob activated carbon at 800°C achieved an efficiency of 97.35% in rhodamine B adsorption after a contact time of 120 minutes, with a 101.01 mg/g adsorption capacity (Setiawan et al., 2025). Similarly, for KOH-activated coconut shells, the adsorption equilibrium of methylene blue was reached at 150 minutes, while Congo red and neutral red were achieved at 120 minutes (Zhang et al., 2018).

Furthermore, a study conducted by Tu et al. (2021) showed that biomass mixing can accelerate the rate and enhance the adsorption efficiency. A 1:1 mixture of sewage sludge and KOH-activated coconut shell mixture exhibited a real synergistic effect in methylene blue removal, reaching 99.5% with an adsorption capacity of 589.37 mg/g. This performance greatly exceeded that of the individual components, a pure sludge reaching only 284.21 mg/g and pure coconut shells with a capacity of 412.73 mg/g. Thus, the balanced biomass mixture effectively maximizes adsorption efficiency.

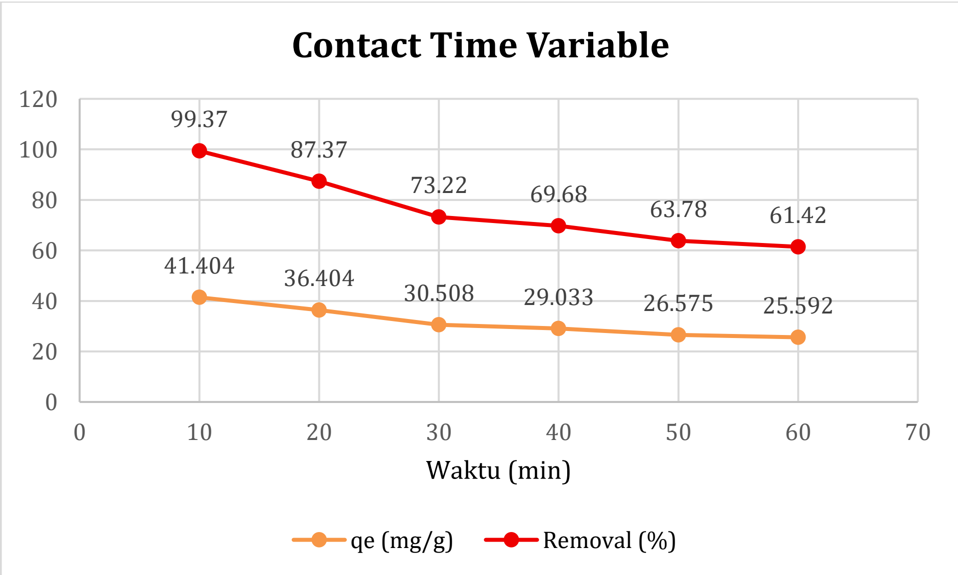

Figure 3. Contact time effect on nitrate removal efficiency (%) and adsorption capacity (qe).

2) Effect of Dosage
In an experiment with 10 min contact time and 40 mg/L initial concentration, in Figure. 4, increasing the adsorbent dose from 30 - 70 mg increases the efficiency of nitrate removal from 50.29 % to 89.45 %. However, the adsorption capacity (qe) actually decreased by 16.73 to 12.79 mg/g. This condition is caused by the larger adsorbent dose, a greater potential for adsorbent mass agglomeration increase. Other studies have also shown that increasing the dose of adsorbents can improve the efficiency of removal, but decrease the adsorption capacity due to active side saturation and particle agglomeration (Shirani et al., 2025).

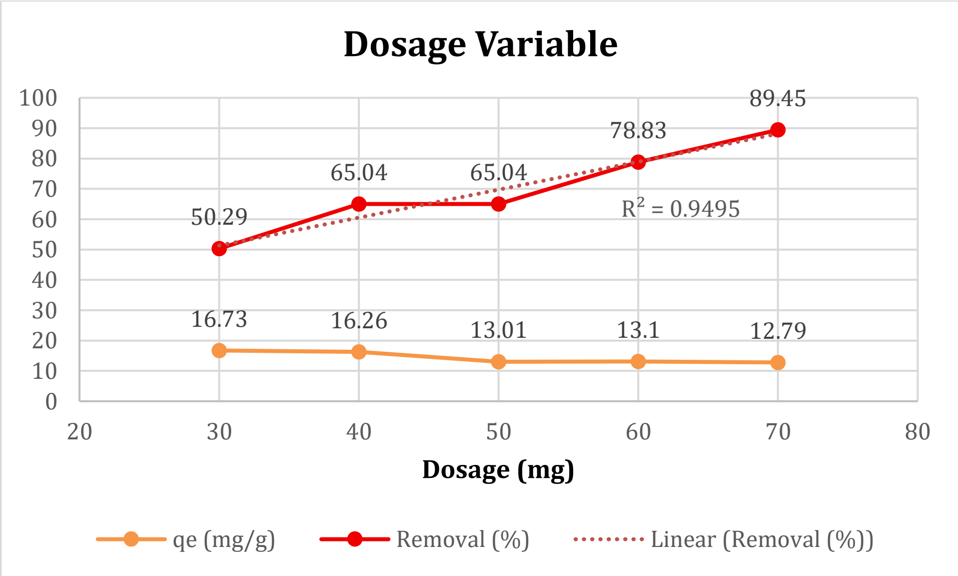

Figure 4 Adsorbent dosage effect on nitrate removal efficiency (%) and adsorption capacity (qe).

CONCLUSION

This study proves that KOH-activated corn cob and coconut shell mixture is effective in removing nitrate ions in waters and is a sustainable solution in agricultural waste treatment. Based on the contact time factor, adsorbent dose, and initial nitrate concentration in *One Factor At a Time* (OFAT) method, the optimal condition was achieved at 10 min contact time, 60 mg adsorbent dose, and 50 mg/L initial nitrate concentration with 99.37% removal efficiency. The results of FTIR and SEM characterization showed the formation of mesopores and macropore structures as well as the active side on the carbon surface after activation with KOH, which improved adsorption performance. Thus, KOH-activated corn cob-coconut cob mixture has high efficiency at low cost for water remediation applications.

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