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Utilizing Coffee Husk Biochar as an Effective Adsorbent for Ammonium Removal in Groundwater

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Abstract

The study investigated the performance of a novel biochar derived from coffee husks (BCFH) and its alkaline-activated product (BCFH-NaOH) for ammonium removal in the aqueous solution. Several batch experiments were conducted with the synthetic solution to determine the adsorption properties of the biochars. At the initial ammonium concentration of 50 mg/L, the optimal dosage for both materials was 10 g/L, while the ideal pH range was 4-9. The equilibration adsorption time on both materials is within 30 minutes, indicating their high practical applicability. Both the pseudo-first order and pseudo-second order kinetic were applied successfully to describe the adsorption kinetics (R^2 >0.95). The isotherms can be defined by both Langmuir and Freundlich models, showing that ammonium removal by biochars is a complex process. BCFH-NaOH showed better performance, with the maximum adsorption capacity reaching 9.97 mg-NH_4/g, compared to 6.64 mg-NH_4/g of BFCH. Finally, BCFH-NaOH was tested with a practical groundwater sample (C_o = 11.5 mg-NH_4/L), achieving a sorption efficiency of up to 80 % while eliminating most of the hardness. These results show that the modified coffee husk biochar could be applied as a low-cost, environmentally friendly adsorbent for ammonium removal.

Keywords: Agricultural waste, coffee husks biochar, ammonium removal, groundwater.

1. Introduction

Vietnam is considered one of the countries with the most significant agricultural economy in South East Asia. By 2021, the total agrarian production in Vietnam is estimated at US$45.26 billion, contributing to 12.56% of the total Vietnam GDP [1]. During the cultivation process, it creates a large amount of agricultural waste, including straws, husks, stubble, leaves, branches, shells, grounds, etc. [2]. Recently, along with the development of the circular economy, many scientists worldwide have paid much attention to recycling these wastes into several secondary products, which benefits humans and reduces the environmental impact [3-5].

Currently, ammonium pollution is one of the major concerns for groundwater quality in Vietnam. The presence of high NH_4^+ concentration in groundwater samples was published by many authors recently [6, 7]. Typically, several methods have been developed for ammonium removal in aqueous solution, including adsorption, ion exchange, membrane separation, ozonation, biological denitrification, and chemical precipitation [8]. Among these methods, adsorption is considered one of the most effective and economical methods due to its numerous advantages, such as simplicity, high selectivity, and reasonable cost for operation and maintenance [9, 10]. Recently, the development of low-cost adsorbents using agricultural by-products for eliminating ammonium has been rising intensively. For instance, Qiang *et al.* [11] synthesized a novel modified biochar utilizing the peanut shell. Under the initial ammonium concentration of 30 mg/L in the dynamic column study, the maximum adsorptive capacity reached 9.02 mg NH_4^+/g. Many other studies were conducted using various agricultural wastes as the raw material sources [12-14].

This study aims to develop a novel biochar derived from coffee husk to effectively eliminate ammonium from the aqueous solution. According to the preliminary test, the coffee husk was selected among several popular agricultural wastes for biochar synthesis; then, the pristine biochar (BCFH) was modified using NaOH to achieve an activated biochar (BCFH-NaOH). A series of batch experiments were conducted with the BCFH and BCFH-NaOH in order to investigate the effect of solution pH, adsorbent dosage, and contact time on the ammonium removal efficiency. The experimental data was also applied to the kinetic and isotherm studies. Eventually, the coffee husk biochar modified with NaOH was tested for the purification of a practical groundwater sample in Hanoi, Vietnam. The results indicate that the biochar synthesized from the coffee husk is a promising adsorbent for eliminating ammonium in groundwater.

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2. Materials and Methods

2.1. Materials

Several agricultural waste materials were collected for this study, including mung bean husks, rubber plat leaves, longan shells, longan seeds, sunflower seed husks, maca nut shells, coffee grounds, and coffee husks. Firstly, they were washed with tap water three times and washed again with distilled water to remove all impurities. After that, the raw materials were dried in an oven to eliminate humidity before implementing the biochar synthesis.

NH_4Cl salt used for the preparation of stock solution was purchased from Merck Chemical Co., Germany. The other chemicals for ammonium analysis in this study, including sodium salicylate ($C_7H_6O_3Na$), trisodium citrate dihydrate ($C_6H_5O_7Na_3.2H_2O$), sodium nitrosopentacyanoferrate (III) dihydrate ($Na_2Fe(CN)_5NO.2H_2O$), sodium hydroxide (NaOH), and sodium dichloroisocyanurate dihydrate ($C_3N_3O_3Cl_2Na.2H_2O$) were all analytical grade.

2.2. Methods

2.2.1. Synthesis of biochar

The raw materials were tightly packed into heat-resistant cups to synthesize the biochar. The pyrolysis process was slowly carried out at 200 – 800 ^{o}C for 1, 2, and 3 hours with a creasing heating rate of 5 ^{o}C per minute. After that, the biochar was rewashed with DI water and dried overnight to remove moisture. Then, they were ground and sieved to obtain the particles with sizes within the range of 0.6 to 1 mm.

The preliminary tests with the obtained biochar were taken place in order to determine the optimal agricultural waste for further experiments. Six different types of agricultural waste were selected as the raw material for the biochar preparation. Specifically, 0.5 grams of each biochar type was put into 50 mL of 50 mg/L NH_4^+ solution in the flasks. The flasks were then shaken at the shaking speed of 100 rpm for 24 hours. After that, the liquid and solid phases were separated by filtering through 0.45 µm filter paper. Then, the remaining ammonium concentration in the solution was analyzed according to the manual spectrometric method (ISO 7150-1:1984) [15]. Additional tests were also ascertained to investigate the optimal conditions for the pyrolysis progress. The temperature was adjusted from 200 to 800 ^{o}C, and the contact time was varied from 1 to 3 hours.

From the preliminary test result, coffee husk was identified as the most appropriate raw material for the biochar. It was then modified by alkaline to obtain the activated material. Specifically, the raw coffee husks biochar was mixed with 1M NaOH solution at a ratio of 2.5 mL NaOH/g for two hours; then, it was pyrolyzed with the same procedure described above. Finally, the modified adsorbent was washed, dried, ground, and sieved to achieve the expected diameter.

2.2.2. Characterization

The morphology of the BCFH-NaOH and the BCFH-NaOH biochar were determined using scanning electron microscopy (SEM, Model JSM-7500F, JEOL Ltd., Japan). To detect the functional group and chemical compositions on the surface of the materials, the Fourier transform infrared (FTIR) analysis was conducted. The specific surface area of the BCFH and the BCFH-NaOH was detected using the BET measurement.

2.3. Batch Experiments

To reveal the capacity of the biochars for ammonium uptake, several batch experiments were carried out. In particular, the model ammonium solution (50 mg/L) was prepared from the stock solution of 1000 mg-NH_4^+/L. A fixed amount of each biochar was added to 100 mL of the artificial solution in a 250 mL Erlenmeyer flask. Then, the adsorption process was implemented according to the procedure in the preliminary test. The ammonium uptake capacity at equilibrium and at time *t* were calculated according to the below mass balance equation:

$$Q_e = \frac{(C_o - C_e)V}{m} \quad (1)$$

$$Q_t = \frac{(C_o - C_t)V}{m} \quad (2)$$

where C_o (mg/L) is the initial concentration of ammonium in the solution; C_t and C_e (mg/L) are the concentration of ammonium in the liquid phase at equilibrium and at time *t*, respectively; Q_e and Q_t (mg/g) are the mass of ammonium adsorbed per gram of biochar at equilibrium and at time *t*, respectively; V (L) is the volume of the solution; and m (g) is the adsorbent mass.

The initial and equilibrium pH was recorded by a Toledo AR10 pH meter (Mettle Toledo). All experiments were repeated thrice, and the standard average was calculated.

2.3.1. Effect of solution pH

The adsorption performance under different pH conditions was studied at a pH range from 2 to 9. Initial pH solutions were adjusted by diluted HCl 0.1 M or NaOH 0.1 M to obtain the expected values. 0.5 grams of biochar was shaken with 100 mL of the ammonium model solution. After the adsorbents were saturated, the effluents were collected and analyzed.

2.3.2. Effect of contact time and kinetic studies

To reveal the effect of contact time on the ammonium uptake of the biochar, 0.5 grams of biochar were shaken in 100 mL of 50 mg/L NH_4^+ solution. The samples were collected periodically from 5 to 30 min for the analysis.

Two empirical models, including the Pseudo-first and Pseudo-second order, were utilized to investigate the sorption kinetics. Equations of these models are introduced below:

Pseudo-first order: $$Q_t = Q_e(1 - e^{-k_1 t}) \quad (3)$$

Pseudo-second order: $$Q_t = \frac{k_2 Q_e^2 t}{1 + k_2 Q_e t} \quad (4)$$

where t (min) is the contact time, k_1 (1/min) and k_2 (g/mg min) are the rate constant of Pseudo-first order and Pseudo-second order expressions, respectively.

2.3.3. Effect of solid/liquid ratio and isotherm studies

In order to determine the optimum adsorbent dosage for the ammonium removal process, different amounts of biochar (equivalent to the solid/liquid ratio of 1.0 – 17.5 g/L) were shaken with 100 mL of synthetic ammonium solution. After 24h, the ammonium concentration in the solution was analyzed, then the percentage of ammonium ions removed could be determined according to the following expression:

$$\text{RE}\ (\%) = \frac{(C_o - C_e)}{C_o} \cdot 100(\%) \quad (5)$$

The data obtained from this test was fitted into two popular isotherm models, including Langmuir and Freundlich, to ascertain the ammonium removal characteristic. The linearized form of these two models is presented in the equations below:

Langmuir expression:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m} C_e + \frac{1}{k_L Q_m} \quad (6)$$

Freundlich expression:

$$l_n Q_e = \frac{1}{n} lnC_e + ln\, k_F \quad (7)$$

where Q_m (mg/g) is the maximum adsorption capacity; K_L (L/mg) and K_F ((mg/g)(L/mg)$^{1/n}$) are the Langmuir and Freundlich constant, respectively; and $1/n$ is the Freundlich intensity parameter.

2.3.4. Performance of BCFH-NaOH biochar in the practical groundwater sample

To determine the practicality of the BCFH-NaOH biochar, a groundwater sample was taken from a household in Ly Nhan district, Ha Nam province, Vietnam. The adsorption test was taken place following the procedure described above. Along with the analysis of NH_4^+ concentration, the content of other cations in the aqueous phase, including Ca, Mg, and Fe, was also measured.

3. Results and Discussions

3.1. Preliminary Test for the Material Selection

It can be observed from Fig. 1 that the sunflower seed husk showed the lowest ammonium removal rate (4.93 %) and adsorption capacity (Q_e = 0.24 mg/g), while the coffee husk achieved the highest efficiency (29.48 %) and NH_4^+ capture amount (Q_e = 1.47 mg/g). Corresponding to the removal efficiency, the adsorption capacity of the biochar-derived coffee husks reached the value of 1.33 mg-NH_4^+/g biochar under the mentioned conditions. Apparently, coffee husk was considered the most applicable material for further studies.

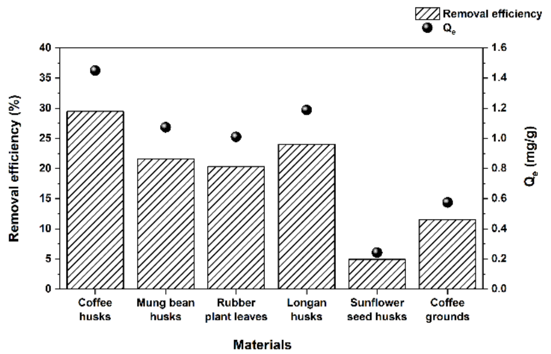

Fig. 1. Performance of biochars synthesized from various types of materials (Experimental conditions: Adsorbent dose = 10 g/L; C_o = 50 mg-NH_4^+/L, pH = 7, pyrolysis temperature = 300 ºC, pyrolysis time = 2h).

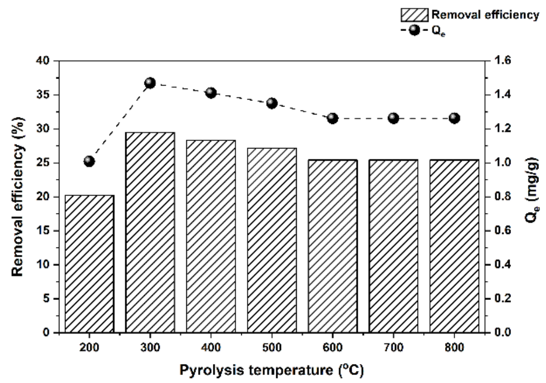

Fig. 2. Effect of pyrolysis temperature (Experimental conditions: Adsorbent dose = 10 g/L; C_o = 50 mg-NH_4^+/L, pH = 7, pyrolysis time = 2h).

Fig. 2 demonstrates the influence of pyrolysis temperature on the efficiency of the synthesized biochars using coffee husk. The percentage of NH_4^+ uptake at 200 ºC was the smallest (20.23%), then the maximum percent (29.48%) was attained when increased the pyrolysis condition to 300 ºC. However, when the synthesis temperature was higher than 300 ºC, the adsorbent's performance decreased slightly. Therefore, the pyrolysis temperature of 300 ºC was highlighted as the most sufficient value for the composition of the biochar using coffee husk material.

The effect of synthesis time is represented in Fig. 3. After two hours of pyrolysis, the material reached the highest removal efficiency (29.48 %) and ion uptake capacity (1.47 mg/g) compared to the corresponding results after 1 h and 3 h.

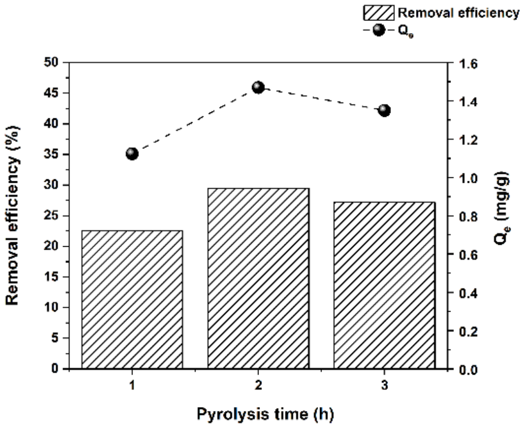

Fig. 3. Effect of pyrolysis time (Experimental conditions: Adsorbent dose = 10 g/L; C_o = 50 mg-NH_4^+/L, pH = 7, pyrolysis temperature = 300 °C).

Eventually, with the view of developing a cost-effective and highly efficient biochar, coffee husk was chosen with the optimum synthesis condition of 2 hours pyrolysis time at 300 °C. As mentioned in the former section, the biochar was also modified using alkaline (1M NaOH) to study the performance of the activated product on the ammonium removal in an aqueous solution. For clarification, the pristine biochar and the activated adsorbent were denoted as BCFH and BCFH-NaOH, respectively.

3.2. Characterization of BCFH and BCFH-NaOH Biochars

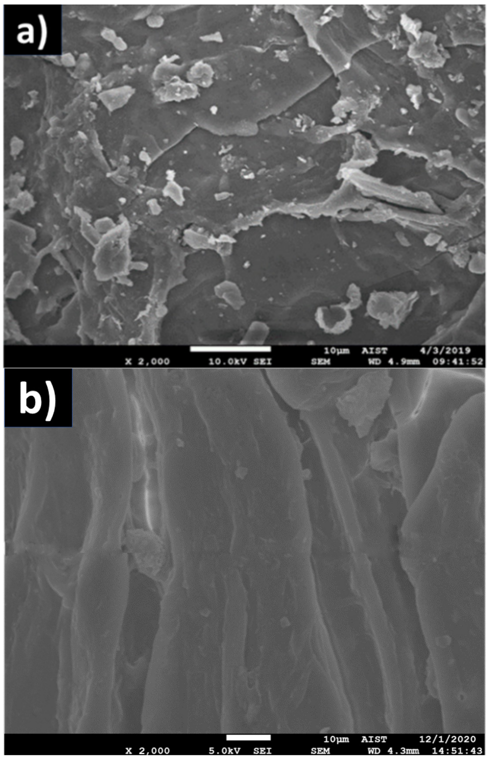

Fig. 4. SEM images of (a) BCFH and (b) BCFH-NaOH.

It can be observed from Fig. 4(a) and 4(b) that the surface of the pristine BCFH and BCFH-NaOH had several folds, roughness and heterogeneity. Generally, for the FTIR measurement at the wavelength from 400÷4000 cm^{-1}, the spectra of the pristine biochar were similar to the modified product. For instance, the band at 1047.35 cm^{-1} for BCFH and 1049.28 cm^{-1} for BCFH-NaOH was attributed to the C-H and C-O stretching vibration [16]. In addition, the spectra at 867÷885 cm^{-1} were assigned to the C-O stretching vibration in the $-OCH_3$ group [17]. However, it can be observed that in FTIR data of BCFH-NaOH, the two spectra at 1406.11 cm^{-1} and 1556.55 cm^{-1}, which were associated with the O-H bending and -COOH stretching vibrations [17], respectively, were expanded compared to the original peaks in BCFH adsorbent. It verifies that the structural properties of the pristine biochar were shifted due to the alkalized step with NaOH. Furthermore, according to the BET measurement, the specific surface area of BCFH biochar was 0.7337 m^2/g, while the corresponding value for BCFH-NaOH biochar was 1.5989 m^2/g. This data also confirms the successful modification of the BCFH-NaOH adsorbent.

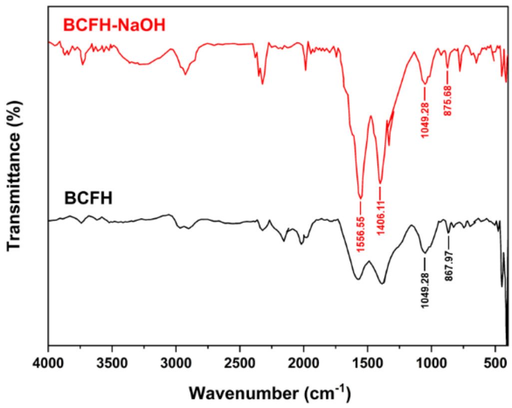

Fig. 5. FTIR spectra of BCFH and BCFH-NaOH.

3.3. Performance of BCFH and BCFH-NaOH Biochars in Batch Studies

3.3.1. Effect of solution pH

The impact of solution pH on the adsorption properties is reflected in Fig. 6. The percentage of ammonium uptake by the original BCFH biochar sustained identically (24.3% - 28.9%) in a wide range of solution pH from 4 to 9. Especially at pH = 5, the BCFH reached the highest ammonium removal rate (28.9%). In addition, the activated BCFH-NaOH biochar has a higher NH_4^+ uptake efficiency (43.4% - 47.8%) within a similar pH range. These results indicate that the biochar derived from coffee husk could be well-operated in acidic and alkaline conditions.

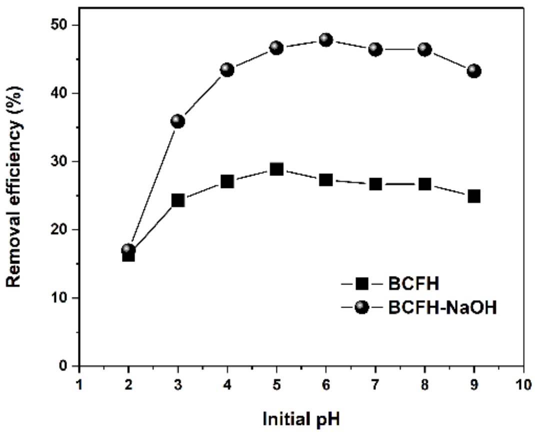

Fig. 6. Effect of initial solution pH (Experimental conditions: Adsorbent dose = 5 g/L; C_o = 50 mg-NH_4^+/L).

The poor NH_4^+ uptake performance at pH < 4 could be explained by the fact that at low solution pH, the presence of H^+ at high concentration competes with NH_4^+ ions for the adsorption by biochar. In addition, the unfavorable electrostatic attraction between the positively charged surface of coffee husk biochars and the NH_4^+ ions also contributed to the negligible uptake amount of ammonium [1]. On the contrary, when the solution pH increased, the deprotonation of the oxygen-based groups (i.e., -COOH) created more negative charge on the biochar's surface, thus contributing to the higher ammonium sorption rate [1].

3.3.2. Effect of contact time and kinetic studies

The effect of contact time on the ammonium uptake was ascertained in Fig. 7. Within the first 15 min, the removal rate increased intensely for both BCFH and BCFH-NaOH biochars. The adsorption process then increased negligibly until reaching the equilibrium state after 30 min. These results show that eliminating ammonium in an aqueous solution using biochar derived from coffee husk could take place rapidly, implying their practicability.

Table 1 presents the kinetic properties of the BCHF and BCHF-NaOH biochars. The experimental data for these two biochars were well-fitted (R^2 >0.95) with the empirical models. Particularly, for the BCFH material, the correlation coefficient for the Pseudo-first order and Pseudo-second order models were 0.975 and 0.993, respectively. For the BCHF-NaOH biochar, the corresponding values were 0.998 and 0.999, respectively. Furthermore, the predicted Q_e for BCFH (2.558 and 2.637 mg/g) and BCFH-NaOH (4.721 and 4.764 mg/g) were in accordance with the factual adsorption capacity at equilibrium (i.e., Q_e = 2.65 mg/g for BCFH and Q_e = 4.76 mg/g for BCFH-NaOH). In other words, it can be concluded that the kinetics of the ammonium removal by the BCHF and BCHF-NaOH biochars could be described correctly using both models.

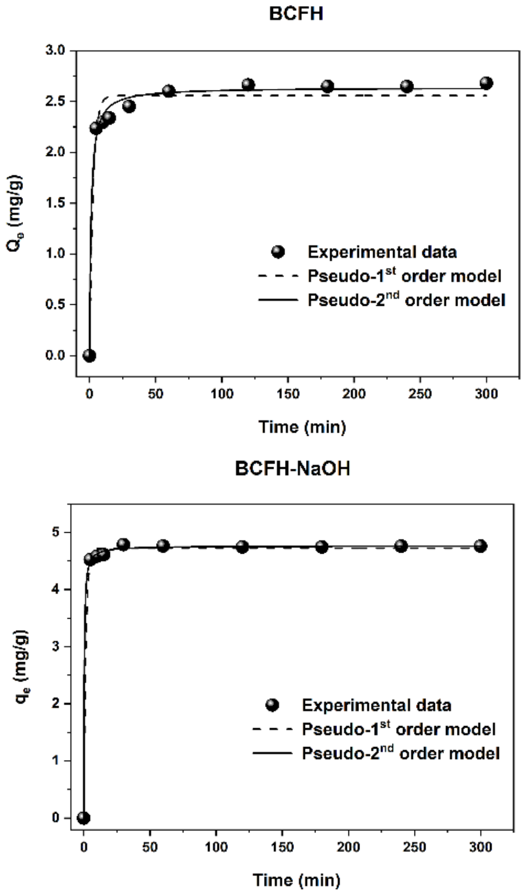

Fig. 7. Experimental data fitted to the Pseudo-1st and Pseudo-2nd kinetic models of a) BCFH and b) BCFH-NaOH (Experimental conditions: Adsorbent dose = 5 g/L; C_o = 50 mg-NH_4^+/L, pH = 7).

Table 1. Kinetic parameters of the Pseudo-first order and Pseudo-second order model.

Materials	Pseudo-1st order			Pseudo-2nd order		
	k_1 (min^{-1})	Q_{e1} (mg/g)	R^2	k_2 (g mg^{-1} min^{-1})	Q_{e2} (mg/g)	R^2
BCFH	0.371	2.558	0.975	0.323	2.637	0.993
BCFH-NaOH	0.622	4.721	0.998	0.696	4.764	0.999

3.3.3. Effect of adsorbent dose and isotherm studies

Fig. 8(a) and 8(b) demonstrate that by increasing the adsorbent dosage from 1 to 10 g/L, the removal rate was increased rapidly. However, when the amount of biochars overcame 10 g/L, the ammonium uptake percentage only rose slightly and reached the equilibrium state. In contrast, the adsorption capacity of both materials decreased notably when the solid/liquid ratio was raised. Considering the removal efficiency and practicability, the adsorbent dose of 10g/L was employed for further experiments.

Fig. 9 displays the correlation between the experimental data and the applied Langmuir and Freundlich isotherm models. It should be noted that both two expressions were well established with the test results obtained from the two biochars (R^2 >0.95). In addition, the Langmuir maximum adsorption capacity of BCFH-NaOH (9.97 mg/g) was marginally superior to the other adsorbent (6.64 mg/g). This could be attributed to the increase of the BET specific surface area and the addition of the functional groups on the surface of the activated biochar, which results in the enhancement in terms of ammonium uptake performance. Table 2 compares the performance of BCFH and BCFH-NaOH in this study with several different biochars derived from agricultural wastes in terms of ammonium capture in the aqueous solution.

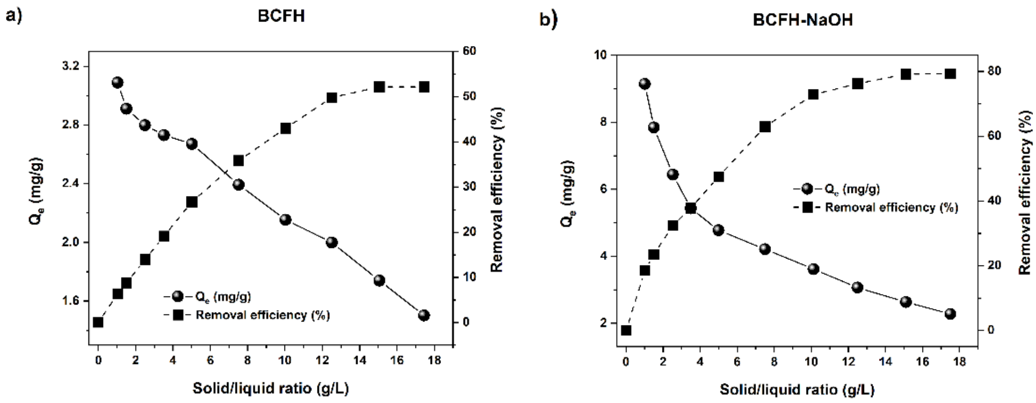

Fig. 8. Effect of solid/liquid ratio on the ammonium removal rate and adsorption capacity of (a) BCFH and (b) BCFH-NaOH (Experimental conditions: Adsorbent dose = 1.0 – 17.5 g/L; C_o = 50 mg-NH_4^+/L, pH = 7)

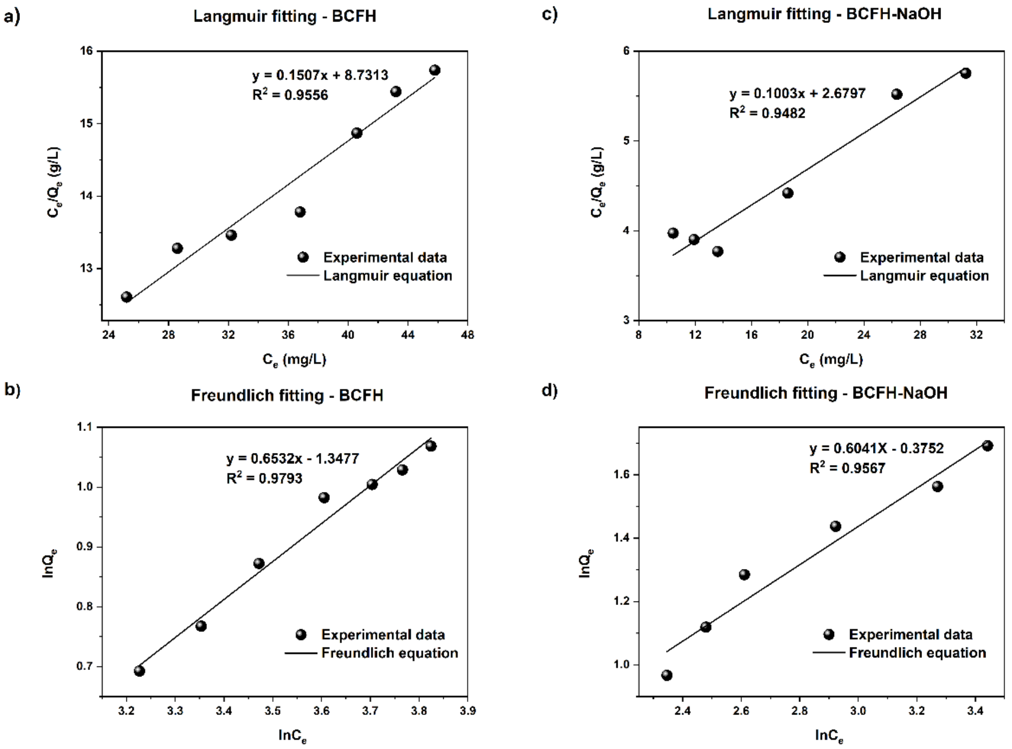

Fig. 9. Experimental data fitted to the Langmuir and Freundlich isotherm models of (a), (b) BCFH and (c), (d) BCFH-NaOH

Table 2. Comparison between the adsorption capacity of the two biochars derived from the coffee husk and other biochars for the ammonium removal in aqueous solution.

Biochar	Pyrolysis condition	Initial NH_4^+ con. (mg/L)	Adsorbent dosage (g/L)	Q_{max} (mg/g)	References
Coffee husk	300ºC, 2h	50	1.0-17.5	6.64	This study
Coffee husk soaked with NaOH	300ºC, 2h	50	1.0-17.5	9.97	This study
Coffee husk	300ºC, 2h	0-50	10.0	1.80	[1]
Coffee husk	350ºC, 1h	10-125	5.0	2.8	[2]
Orange peel	300ºC, 2h	40	10.0	4.71	[3]
Pineapple peel	300ºC, 2h	40	10.0	5.60	[3]
Peanut shell	500ºC, 2h	10-100	2.0	3.83	[4]
Corn straw	400ºC, 1.5h	50	4.0	2.3	[5]

3.3.4. Removal efficiency of BCFH-NaOH biochar with practical groundwater sample

Table 3 summarizes the test outcome of BCFH-NaOH material for the groundwater sample collected from a household in Hanam province, Vietnam. Regarding NH_4^+ uptake performance, the BCFH-NaOH biochar achieved a removal rate of approximately 78% at the initial NH_4^+ concentration of 11.56 mg/L (Effluent NH_4^+ concentration was 2.56 mg-NH_4/L). In addition, it is apparent that this biochar also effectively eliminated the hardness in groundwater. For instance, after the adsorption process, the final Ca, Mg, and Fe concentrations were lower than 0.01 mg/L. It indicates that this modified biochar derived from the coffee husk is a promising adsorbent for ammonium treatment in contaminated groundwater.

Table 3. Performance of biochar BCFH-NaOH with a groundwater sample in Ha Nam province, Vietnam.

Parameter	Sample	
	Initial	Effluent
Ca (mg/L)	20.4	<0.01
Mg (mg/L)	11.6	<0.01
Fe (mg/L)	<0.05	<0.01
NH_4^+ (mg/L)	11.56	2.56

4. Conclusion

In this article, biochar derived from coffee husk was successfully fabricated. The pristine biochar, BCHF, was also modified with alkaline to improve the adsorption performance. The batch study showed that the optimal solution pH condition for the ammonium removal using the original biochar and the activated product was 4-9. Furthermore, the optimum adsorbent dosage for both biochars was 10 g/L. Either Langmuir or Freundlich's linearized equation could be well applied to describe the isotherm properties of these two biochars. Interestingly, the Langmuir Q_{max} for BCFH-NaOH was 9.97 mg-NH_4^+/g, around 50 % higher than the corresponding value for the neat biochar (6.64 mg-NH_4/g). In the kinetics study, the adsorption process could be taken place efficiently and reached an equilibrium state within 30 minutes for both materials. The two models, including the Pseudo-first order and Pseudo-second order models, could be applied to explain the kinetics of the ammonium removal process using both the biochars with a relatively high correlation coefficient (R^2 >0.95). Eventually, in the practical study with the groundwater sample, the biochar BCFH-NaOH exhibited the efficiency to simultaneously eliminate ammonium and hardness in the complex solution, which confirms its practicability for ammonium treatment in groundwater.

Acknowledgments

Credit authorship contribution statement. Dai Quyet Truong: Methodology, Conceptualization, Writing manuscript. Van Phu Pham: Conceptualization, Data analysis. Le Minh Tran: Supervision, Conceptualization, Review and Editing. Tien Vinh Nguyen: Supervision, Conceptualization, Review and Editing.

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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