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RESEARCH ON THE RECYCLING OF WASTE ACTIVATED SLUDGE AS AN ADSORBENT MATERIAL FOR AMMONIUM REMOVAL IN WASTEWATER

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Abstract. Waste activated sludge is an abundant byproduct of wastewater treatment plants (WWTPs) considered to be a secondary pollutant due to its large amount and unmanageable production. Recently, the preparation of sludge-derived activated carbon or activated sludge as a novel adsorbent has been reported, which is also considered as a new alternative method for sludge treatment without secondary pollution. In this study, sludge-derived activated carbon or activated sludge was fabricated from the sludge generated by the WWTP of an industrial brewery through a pyrolysis technique to achieve a new adsorbent for ammonium removal. The sludge-based biochar samples possessed more porous structure, larger specific surface area and pore size compared to the dried sludge sample. Batch adsorption experiments were conducted to investigate the removal efficiency and adsorption capacity for ammonium. The results showed that the operating conditions for ammonium adsorption were optimized at BS400 (400 °C - pyrolyzed sludge) dosage of 20 g/L, initial ammonium concentration of 30 mg/L at pH 6 for a total contact time of 120 min. The experimental data best fitted the Langmuir isotherm, while the kinetics followed the pseudo-second-order model. The column adsorption showed that 10 g of BS400 could maintain 375 mL and 1050 mL of 20 mg/L and 10 mg/L NH_4^+ solutions to meet the NH_4^+ threshold in the National Technical Regulation on Industrial Wastewater (QCVN40:2021/BTNMT) with the adsorption capacity of 0.642 and 0.784 mg/g, respectively.

Keywords: activated sludge, ammonium adsorption, wastewater treatment, biochar, pyrolysis.

Classification numbers: 3.3.2, 3.4.2.

1. INTRODUCTION

Ammonium ion (NH_4^+) is the most dominant form of widespread nitrogen pollution in the water environment and its toxicity causes serious environmental problems, which has been widely reported [1]. This pollution enters the environment via various sources such as municipal and animal feedlots wastewater, leachate, agriculture activities, and so on [2, 3]. High concentrations of NH_4^+ in water can cause eutrophication and stimulate algal growth, reducing dissolved oxygen levels, threatening aquatic life, and causing undesirable changes in ecosystems

[3, 4]. Especially, ammonium ions are toxic to some kinds of fish even at concentrations as low as 3 ppm [5]. Moreover, ammonia (NH_3) can be formed by hydrolyzation of ammonium (NH_4^+) and they are easily interchangeable depending upon the pH condition of the water. As reported, ammonia is much more toxic than ammonium [6], NH_3 gas can also cause corrosion phenomena and bad odors. However, in natural water, NH_4^+ has much higher concentration than NH_3 due to neutral pH condition. Hence, for ensuring the quality of the water environment, ammonium needs to be removed.

Accordingly, many methods have been used and developed to remove ammonium from wastewater such as physical, chemical, and biological methods including chemical precipitation [7], chlorination [8], supercritical water oxidation [9], reverse osmosis [10], and biological technology [7, 11]. However, there are various disadvantages and limitations of these technologies that exist, consisting of high cost, low removal efficiency, and presence of byproducts that can be new pollutants. Among these technologies, ion exchange and adsorption can be promising techniques due to many advantages such as high removal efficiency, low cost, easy operation, and having a large number of adsorbents that are available depending on the intended use and treatment of pollutants nowadays [9]. Over the past few decades, WWTPs have exhibited good applications in improving water quality and providing water for multiple uses such as living activities, breeding, irrigation, etc. However, waste activated sludge is an abundant by-product of WWTPs, which is considered a new pollutant due to the inclusion of harmful inorganic/organic substances as well as biological hazardous compounds [12]. To this day, the proper disposal of these sludges remains a difficult problem because of the complex and high-cost techniques, the most common methods such as landfill disposal and incineration [13] are gradually rejected due to adverse impacts on the environment. The unmanageable sludge production can be a major challenge that has attracted the attention of researchers to find out a reliable and sustainable solution. Recently, many researchers have reported the use of sewage sludge to fabricate new adsorbent materials such as activated carbon [14] and biochar [15] as an alternative solution to conventional disposal methods, this topic has received a lot of attention.

In this study, due to its abundance and high carbon content, activated sludge-based biochar was fabricated from the sludge of the WWTP in an industrial brewery through a pyrolysis technique to achieve a new adsorbent for ammonium removal. The influence of carbonization temperature on the porosity and surface area of the obtained samples as well as their adsorption capacity were studied. The effects of adsorbent dosage, solution pH, contact time, and initial concentration of NH_4^+ on the adsorption by sludge-based biochar material were also investigated.

2. MATERIALS AND METHODS

2.1. Materials

The wet compressed activated sludge was collected from the WWTP of Hanoi Beer Alcohol and Beverage company (HABECO). The wet sludge was sliced and ground into small particles before being dried at 110 ^oC for 4 hours in an oven to completely remove the humidity (SP). Around 100 g of dried ground sludge was compacted in a steel cup, and pyrolyzed in a carbonization furnace at 300 (BS300), 400 (BS400), and 500 ^oC (BS500) with a heating rate of 5 ^oC/min under an inert environment by passing 5 L/min of N_2 gas into the furnace chamber. The target temperature was kept constant for 2 hours before the furnace was turned off. The obtained biochar was then crushed and sieved into the particle size of 0.25 to 0.5 mm.

2.2. Characterization of materials

The sludge particles and sludge-based biochar were degassed at 200 °C for 12 h under vacuum before the surface area and porosity measurements. The Brunauer-Emmett-Teller (BET) and Barret-Joyner-Halenda (BJH) methods were used to determine the surface area and calculate the total pore volume and average pore width using a TriStar II Plus (Micromeretics Instrument Corporation, United States).

The surface morphology was determined using a Hitachi S3400 scanning electron microscope which was operated at 5.0 kV.

2.3. Batch adsorption experiments

NH_4^+ solution was prepared by dissolving ammonium chloride (Merck, Germany) with distilled water to selected concentration. Batch experiments were conducted by allowing a fixed amount of adsorbents to come into contact with 50 mL of 20 mg/L NH_4^+ solution for a certain period inside a 100 mL conical flask placed in an incubated shaker (Lab Companion, United States) at 150 rpm. The first batch was conducted to study the effect of pyrolysis temperature on NH_4^+ adsorption capacity of each sludge-based biochar under experimental conditions: pH 6, contact time of 4 hours, adsorbent dosage of 10 g/L in 50 mL of 20 mg/L NH_4^+ solution. Adsorbent dosage was also tested in the range of 0.2 g/L to 40 g/L (pH, volume, concentration of NH_4^+ solution and reaction time of 4 hours were kept constant) to determine the optimum amount of the chosen material for NH_4^+ adsorption. The solution pH was set at different values from 2 to 10 to study its influence on the NH_4^+ adsorption with the optimum adsorbent dosage (volume, concentration of NH_4^+ solution and reaction time of 4 hours were kept constant). The kinetics of NH_4^+ adsorption was evaluated at different contact times ranging from 15 to 240 min at optimum adsorbent dosage and pH solution, here a volume of 50 mL and an initial concentration of 20 mg/L of NH_4^+ solution were maintained. The last batch was to examine the effect of initial concentration of ammonium on adsorption, different initial concentrations of NH_4^+ from 5 to 150 mg/L were applied with the optimum adsorbent dosage and solution pH determined in previous batch experiments. A pH meter (HI 2211, Hanna Instrument, United State) was used for all pH measurement. The suspensions were filtered and the filtrates were analysed for NH_4^+ by manual spectrometric method according to TCVN 6179-1:1996 using a UV-VIS Double Beam PC Scanning Auto Cell spectrophotometer (UVD-3200, Labomed Inc, United State) [16]. The amount of NH_4^+ adsorption at equilibrium, q_e (mg/g), was calculated according to the equation below:

$$\boldsymbol{q_e = \frac{(C_o - C_e).V}{m}} \tag{1}$$

where C_o is the initial concentration of NH_4^+ (mg/L), C_e is equilibrium concentration of NH_4^+ (mg/L), V is the volume of the solution (L), and m is the adsorbent mass (g). The NH_4^+ removal efficiency ($\boldsymbol{\eta}$) was calculated by the following equation:

$$\boldsymbol{\eta}\,(\%) = \boldsymbol{\frac{(C_o - C_e)}{C_o} \times 100} \tag{2}$$

The data obtained from the experiments were used to model the adsorption of ammonium using two typical adsorption models, Langmuir (eq. 3) and Freundlich (eq.4) adsorption isotherms [17, 18]:

$$\boldsymbol{q_e = \frac{q_m K_L C_e}{1 + K_L C_e}} \tag{3}$$

where q_m is the maximum NH_4^+ adsorption capacity (mg/g) and K_L is the Langmuir constant (L/mg) relating to the energy of adsorption.

$$q_e = K_F \times C_e^{1/n} \quad (4)$$

where K_F is the Freundlich constant (mg/g)(L/mg)$^{1/n}$ and n is the dimensionless Freundlich constant.

When the experimental data fitted well the Langmuir model, the separation factor (R_L) needs to be calculated. R_L values could be obtained using the following equation:

$$R_L = \frac{1}{1+k_L C_o} \quad (5)$$

where C_o is the initial concentration of adsorbate (mg/L), and k_L is the Langmuir equilibrium constant (L/mg). The separation factor R_L shows whether the adsorption is irreversible ($R_L \approx 0$), favorable ($0 < R_L < 1$), linear ($R_L = 1$), or unfavorable ($R_L > 1$).

The pseudo-first order (eq. 6) and pseudo-second order (eq. 7) equations were used to calculate the kinetics of adsorption [19]:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (6)$$

where k_1 is the rate constant for the pseudo-first order equation (1/min), q_e is the amount of ammonium (mg/g) adsorbed at equilibrium, and q_t is the uptake of ammonium (mg/g) at time *t* (min).

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (7)$$

where k_2 is the rate constant for the pseudo-second order equation (g/mg.min).

2.4. Column experiments

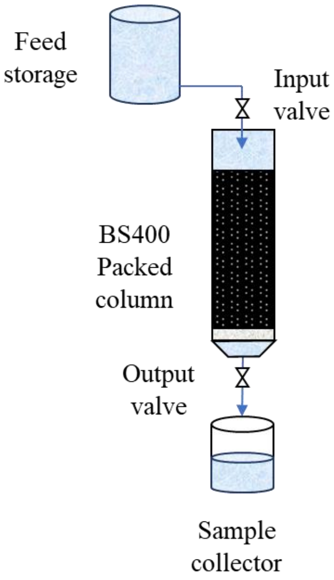


Figure 1. Diagram of column experiments.

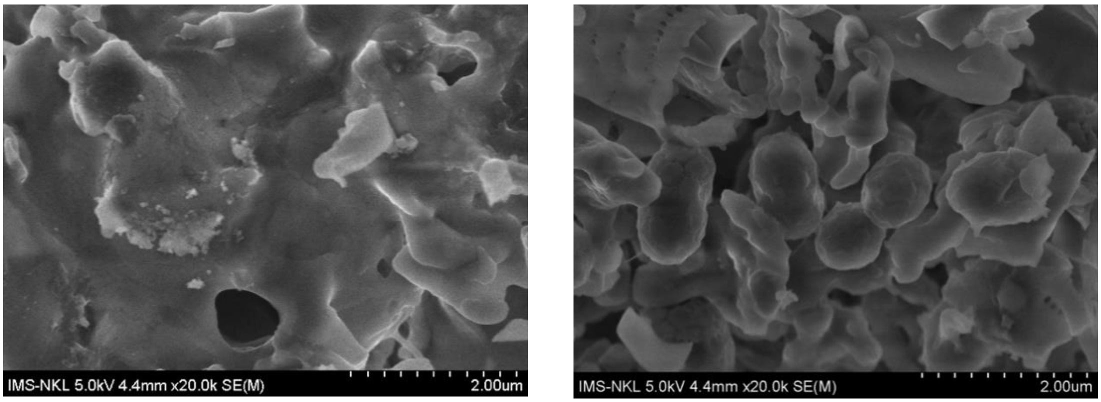
A 25 mL medical syringe (2 cm in diameter and 10 cm in height) packed with 10 g of BS400 was used to simulate a filter column for this experiment. A solution containing 10 or 20 mg/L of NH_4^+ was passed through the column at a flow rate of 5 mL/min in the downflow direction controlled by input and output liquid valves. At the bottom of the column, a thin layer of cotton filer was placed to prevent the material from being carried away by the flow. Before the NH_4^+ solution was fed to the column, the column was prefilled with distilled water during the packing of material to prevent trapped bubbles. The samples were collected after 10 minutes since the feeding of NH_4^+ started.

3. RESULTS AND DISCUSSION

3.1. Characteristics of the sludge particle (SP) and sludge-based biochar adsorbents

Table 1. BET surface area (S_{BET}) and pore volume (V_P) of dried sludge and sludge-based biochar materials.

Sample	**S_{BET} (m^2/g)**	**V_P (cm^3/g)**	**Average pore width (nm)**
SP	41.0341	0.084975	8.20180
BS300	38.7849	0.144194	14.25887
BS400	91.2630	0.259401	11.06249
BS500	75.4786	0.230900	11.82954



(a) *(b)*

Figure 2. SEM images of (a) dried sludge particle (SP) and (b) BS400 material.

The surface and structure characteristics of sludge particle and the obtained biochar are presented in Table 1. The results indicated that the pyrolysis process at high temperature can greatly exhibit surface area, pore size and pore width. The 400 °C-pyrolyzed sludge (BS400) possessed a specific surface area of 91.26 m^2/g, which was more than twice that of the dried sludge particle (SP). The S_{Bet}, however, was slightly smaller for BS500 even though it was processed at higher temperature. This pattern can be explained that the organic content was completely oxidized and volatilized from the sludge under high temperature condition, leaving large porosity in the particle structure. However, excessively high temperature may result in deterioration of biochar structure leading to lower S_{Bet} and V_p of biochar. The pattern found in this study was in good agreement with the results of Florez *et al*. [20]. According to which, palm shell biochar was optimally improved in terms of S_{Bet} when pyrolysis was performed at 650 °C, while pyrolysis at 750 °C resulted in smaller pore size and surface area due to deteriorated structure. Scanning electron micrograph indicated that there was a significant improvement in the morphology of sludge particle surface after pyrolysis process (Figure 2). Under the effect of high temperature pyrolysis, the surface of BS400 became extensively scabrous with the existence of small pores.

3.2. Comparison of adsorption capacity between sludge-based biochar materials

The comparison of NH_4^+ adsorption capacity between SP and sludge-based biochar fabricated at different pyrolysis temperatures is presented in Figure 3. It can be observed that pyrolysis played an important role in exhibiting the adsorption capacity of the sludge. The q value firstly increased from 0.139 to 0.375 mg/g after the sludge was processed at 300 °C, then it was greatly improved (reaching 0.645 mg/g) at a pyrolysis temperature of 400 °C. The enhancement after pyrolysis was the result of the enlargement of pore size and specific surface area. When the temperature reached 500 °C, the q value was recorded as 0.544 mg/g, which was slightly lower than the value at 400 °C. This pattern was due to the change in pore size and surface area of the adsorbent as a result of adjusting pyrolysis temperature. Therefore, the BS400 sample was chosen for further investigations.

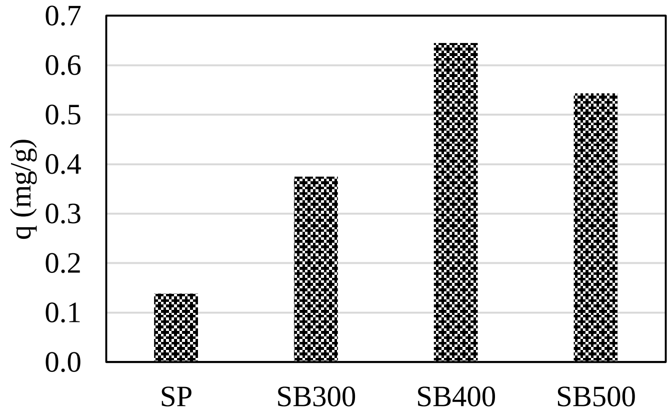


Figure 3. NH_4^+ adsorption capacity of sludge-based biochar materials.

3.3. Effect of adsorbent dosage

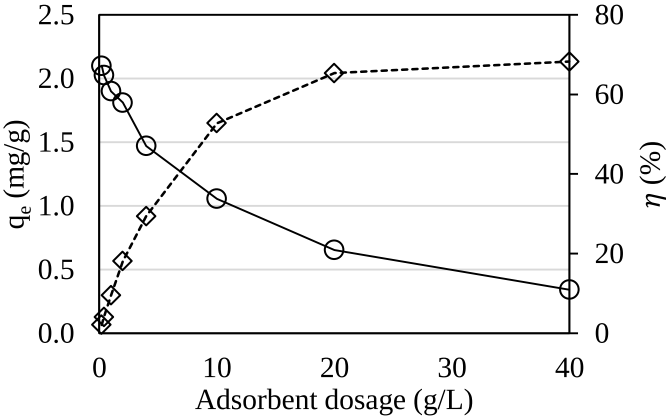


Figure 4. Effect of BS400 dosage on its NH_4^+ adsorption capacity and removal efficiency (○: adsorption capacity, ◊: removal efficiency).

The effect of adsorbent dosage on NH_4^+ adsorption capacity and removal efficiency of BS400 sample was investigated. Figure 4 shows that the q value decreased slightly (from 2.03 to 1.81 mg/g) as the adsorbent dosage increased from 0.2 to 0.4 g/L. The adsorption capacity of the material decreased significantly to 1.06 mg/g as the dosage increased to 10 g/L. The q value continued to go down with higher dosage and reached the minimum value of 0.34 mg/g at the dosage of 40 g/L. In contrast, the removal efficiency of NH_4^+ had the opposite graph compared to the adsorption capacity of BS400 when increasing the adsorbent dosage. The η value was boosted significantly from only 2.1 % to 65.4 % of NH_4^+ removal as the dosage increased from 0.2 to 20 g/L. The removal efficiency then slightly increased and reached the maximum value of 68.3 % as the dosage reached 40 g/L. In summary, it can be seen that with increasing the adsorbent dosage a higher percentage of NH_4^+ removal efficiency could be achieved but the adsorption capacity decreased due to the excessive amounts of adsorbent leading to the unsaturation of the adsorbent binding sites. On the other hand, it might be the reason that the agglomeration or stacking of the adsorbent sites reduced the overall surface area of the adsorbent [21], resulting in a slight increase in removal efficiency at higher dosages used (2.9 % increased from dosage used of 20 to 40 g/L). Therefore, 20 g/L was considered as the optimum dosage of BS400 to use in subsequent experiments.

3.4. Effect of pH

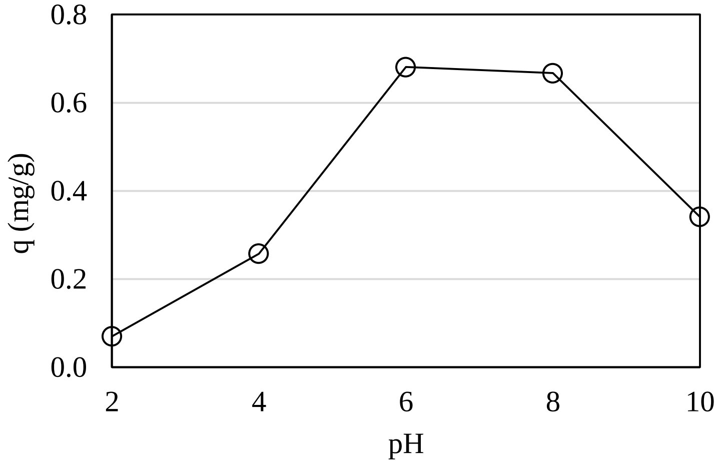


Figure 5. Effect of pH level on the NH_4^+ adsorption capacity of BS400.

The solution pH is an important parameter that can significantly influence the adsorption capacity of the adsorbent. The influence of pH on the adsorption of NH^{4+} by BS400 is illustrated in Figure 5. The results showed that BS400 exhibited the highest q value of 0.68 mg/g at pH 6, which was consistent with a previous research that the maximum adsorption amount for NH_4^+ was usually obtained in the pH range from 6 to 9 [22]. Clearly, in acidic media, H^+ ions formed from pH adjustment process would compete with NH_4^+ for adsorption on BS400 adsorbent binding sites [23], resulting in a relatively low q value at lower pH conditions and it was found that the lowest q value was 0.07 mg/g at pH 2. The adsorption capacity of BS400 increased sharply with increasing the solution pH from 2 to 6 but then decreased when the solution became strong basic. At higher pH (pH > 9), uncharged NH_3 gas was formed by the hydrolyzation of NH_4^+ and could be easily volatilized into the atmosphere [24], which resulted in low adsorption capacity of the adsorbent.

3.5. Kinetics of batch NH_4^+ adsorption

The time and mechanism of NH_4^+ adsorption on BS400 depend on the physical and/or chemical characteristics of the adsorbent as well as on the mass transport process, then the pseudo-first-order and the pseudo-second-order kinetic models were used to find the best fit model and suitable parameters for the adsorption process. The correlation coefficients and other parameters of both kinetic models were calculated and presented in Table 2.

It is easily observed that the pseudo-second-order kinetic model could be identified as the most fitted model for NH_4^+ adsorption by BS400. As seen from Table 2, the correlation coefficient of the pseudo-second-order model (R^2=0.997) was higher than that of the pseudo-first-order model (R^2=0.906), and the calculated equilibrium adsorption capacity (q_e = 0.72 mg/g) was also close to the experimental value of 0.67 mg/g. These results indicated that the ammonium adsorption kinetics by BS400 followed the pseudo-second-order model. Zhang *et al*. (2018) reported that the Lagergren pseudo-second-order model was the optimum model for the adsorption kinetics of NH_4^+ on magnetic excess sludge [25]. The sorption behaviors of NH_4^+ onto H_2O_2 modified-hydrochar derived from paper waste sludge were also best described by the pseudo-second-order kinetic model as shown by Nguyen *et al*. (2021) [26].

Table 2. Batch kinetic model parameter values and coefficient of determination (R^2) of models fitting the data for the adsorption of NH_4^+ on BS400 at pH 6 (with adsorbent dosage of 20 g/L and NH_4^+ concentration of 20 mg/L).

Adsorbent	**Pseudo-first order (PFO)**			**Pseudo-second order (PSO)**		
	q_e mg/g	k_1 min^{-1}	R^2	q_e mg/g	k_2 g/mg.min	R^2
BS400	0.365	0.019	0.906	0.721	0.096	0.997

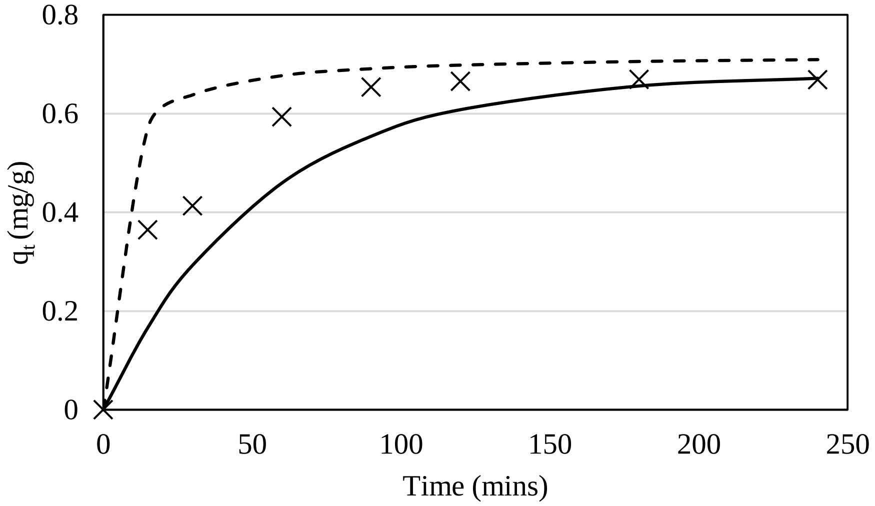


Figure 6. Batch kinetic data for NH_4^+ adsorption on BS400 (×: experimental data, solid line: PFO prediction model, dashed line: PSO prediction model).

3.6. Batch NH_4^+ adsorption at equilibrium

To study the effect of the initial concentration of NH_4^+ in solution on the adsorption capacity, the adsorption activity of BS400 was investigated with the initial concentration of NH_4^+ varying in the range of 5 - 150 mg/L (here, the solution pH of 6 and the reaction time of 120 min were maintained), and the results obtained were presented in Figure 7. It could be easily observed that increasing the initial concentration resulted in higher adsorption capacity but lowering the removal efficiency of the adsorbent. The q value of BS400 increased significantly and nearly tripled (from 0.25 to 0.69 mg/g) when NH_4^+ concentration increased from 5 to 30 mg/L, while the η value was halved from 98.2 to 46.0 %. As the initial concentration increased to 50 mg/L, the adsorption capacity dropped slightly to 0.65 mg/g according to the saturation of the adsorbent and then marginally fluctuated at higher concentrations, the q value reached a peak of 0.75 mg/g at an initial concentration of 150 mg/L. In contrast, the removal efficiency of the adsorbent continued to drop at higher initial concentrations. The η value reached a minimum of 9.9 % at C_o of 150 mg/L. It could be found that the removal efficiency decreased as a result of increasing the initial concentration while the adsorbent had already reached the saturated state. Thus, 30 mg/L was determined as the critical concentration of NH_4^+ at pH 6 to be efficiently removed by 20 g/L BS400.

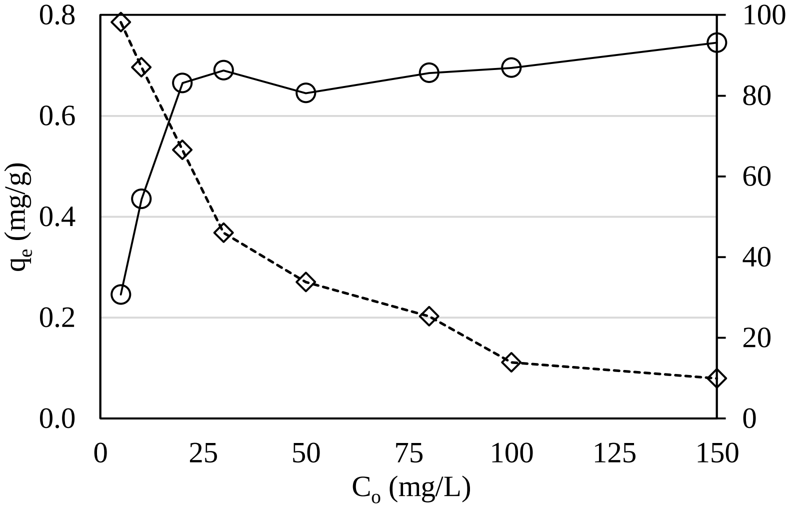


Figure 7. Effect of NH_4^+ initial concentration on the adsorption capacity and removal efficiency (○: adsorption capacity, ◊: removal efficiency).

Figure 8 demonstrated the adsorption isotherms with the Langmuir and Freundlich models to further comprehend the NH_4^+ adsorption mechanism on BS400. It can be seen from the data in Table 3 that the experimental data better fitted the Langmuir model with a higher correlation coefficient ($R^2 = 0.99$) compared to the Freundlich model ($R^2 = 0.88$). The adsorption capacity value calculated from the Langmuir model ($q_m = 0.73$ mg/g) was also close to the experimental data (q = 0.75 mg/g). These results showed that NH_4^+ adsorption mechanism on BS400 followed the Langmuir model, and the adsorption occurred through the monolayer adsorption mechanism and the adsorbent surface was homogeneous [27]. The theoretical NH_4^+ adsorption capacity of BS400 was lower than most of the adsorbents from previous reports. Zhang *et al*. (2018) [25] demonstrated the maximum ammonium adsorption capacity of 1.79 mg/g of the magnetic excess sludge. Hence, further modification of BS400 needs to be studied to enhance the adsorption capacity of the adsorbent.

Table 3. Langmuir and Freundlich model parameters and coefficient of determination (R^2) of the models fitting the data for NH_4^+ adsorption on BS400 at pH 6.

Adsorbent	**Langmuir model**			**Freundlich model**		
	q_m (mg/g)	K_L (L/mg)	R^2	K_F (mg/g)(L/mg)$^{1/n}$	n	R^2
BS400	0.73	0.51	0.99	0.4	6.99	0.88

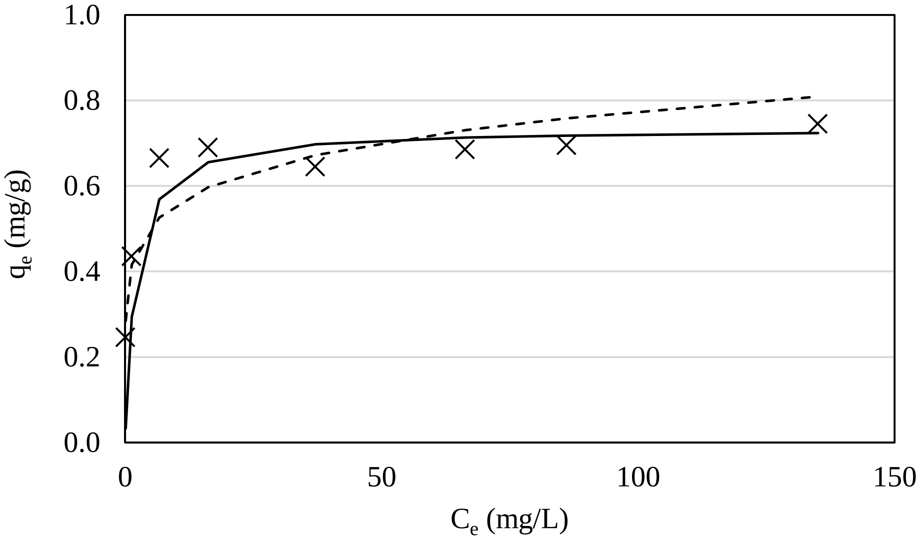


Figure 8. Batch equilibrium data for the adsorption of NH_4^+ on BS400 at pH 6 and the fit of experimental data (×) with Langmuir (solid line) and Freundlich (dashed line) models.

With NH_4^+ concentration varying from 5 to 150 mg/L, the R_L value obtained was in the range of 0.25 – 0.01, which was consistent with the favorable adsorption process. With Freundlich model, 1/n is related to multilayer adsorption capacity and adsorption intensity, it was found that the Freundlich model also well fits the NH_4^+ adsorption with the obtained 1/n value of 0.14, showing that the adsorption process was favorable. These results have also been reported in other previous studies [28, 29].

3.7. Column adsorption results

The curves from column experiments for the NH_4^+ adsorption at the initial concentrations of 10 and 20 mg/L are presented in Figure 9. The results showed that BS400 can treat NH_4^+ wastewater at 10 mg/L and 20 mg/L, complying with the standards for NH_4^+ in the National Technical Regulation on Industrial Wastewater (QCVN 40:2021/BTNMT) [30]. According to the results, 10 g of BS400 biochar could be used for keeping 375 mL and 1050 mL of 20 mg/L and 10 mg/L NH_4^+solutions, respectively, to meet the QCVN 40:2021/BTNMT guideline. The column NH_4^+ adsorption capacity of BS400 at the breakthrough point towards 10 mg/L and 20 mg/L of NH_4^+ were 0.642 and 0.784 mg/g, respectively. These values were similar to the q_m of 0.730 mg/g obtained from the Langmuir model.

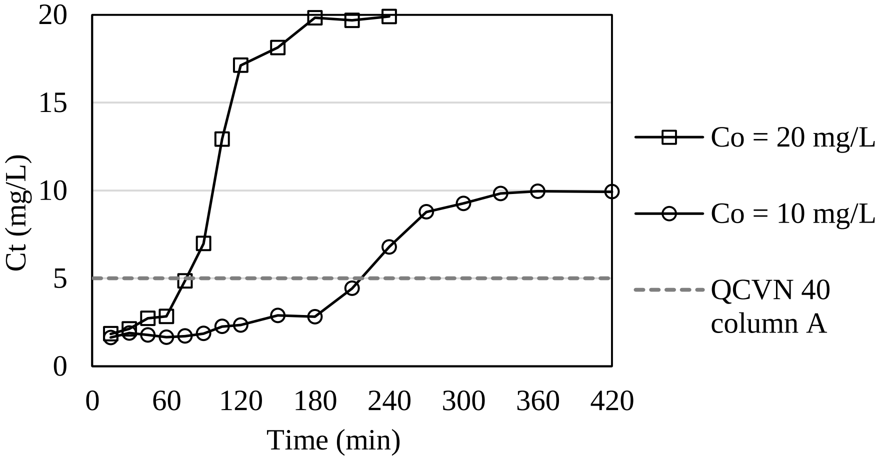


Figure 9. Breakthrough curves for NH_4^+ adsorption on BS400 at different initial concentrations.

4. CONCLUSIONS

The sludge-based biochar materials were successfully fabricated and evaluated for their adsorption potential to remove ammonium from wastewater via batch experiments. The NH_4^+ adsorption on BS400 was better fitted with the Langmuir isotherm with a high correlation coefficient (R^2=0.99) compared to the Freundlich isotherm. The maximum NH_4^+ adsorption capacity of BS400 was calculated as 0.73 mg/g according to the Langmuir isotherm model. The pseudo-second-order model was the optimum model to describe the kinetics of NH_4^+ adsorption on BS400 ($R^2 = 0.997$). The column adsorption showed that 10 g of BS400 could maintain 375 mL and 1050 mL of 20 mg/L and 10 mg/L NH_4^+ solutions to meet the guideline of QCVN 40 with the adsorption capacities of 0.642 and 0.784 mg/g, respectively. This research showed that sludge-based biochar has relatively good potential as a low-cost recycled adsorbent to remove ammonium from water bodies.

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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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