Paper:
Yaser, A., Yusof, L., Ibrahim, A. & Hilal, N. (2018). Adsorption of Ammonia Nitrogen by using Jackfruit (Artocarpus heterophyllus) Seeds: Batch and Fixed-bed Column Studies. *Current Environmental Engineering, 05*
http://dx.doi.org/10.2174/2212717805666180809094826

Adsorption of Ammonia Nitrogen by using Jackfruit (*Artocarpus heterophyllus*) Seeds: Batch and Fixed-bed Column Studies

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ARTICLE INFO

ABSTRACT

The performance of jackfruit (*Artocarpus heterophyllus*) seed adsorbent for ammonia nitrogen (NH_3-N) removal from aqueous solution was examined through batch and continuous bed column experiments. The effects of sodium chloride (NaCl) and lignin concentration on the adsorption process were evaluated. The results revealed that the adsorption performance gradually decreased upon the addition of NaCl and lignin in the solution. Fixed bed column experiments showed that maximum removal of ammonia nitrogen was obtained at an influent concentration of 100 mg/L, bed height of 10 cm and lowest inlet flow rate of 17 mL/min. Meanwhile, desorption studies were carried out at different pH and highest desorption capacity of jackfruit seed adsorbent was 0.42 mg/g. This study suggests that jackfruit seed is a promising adsorbent for the recovery process of ammonia nitrogen.

Keywords: Ammonia-nitrogen, Jackfruit seeds, Adsorption, Fixed-bed

1. Introduction

Ammonia nitrogen is one of the most common and toxic species of nitrogen found in water bodies and the main sources of this pollutant comes from agricultural run-off and industrial wastewater such as detergent manufacturing and mineral processing industry [1]. An excess amount of it in the environment could lead to a serious nature distortion as it may causes eutrophication and toxicity to aquatic organisms [2]. Thus, the treatment process of NH_3-N contaminant from water ways is essential for diminishing serious impact to environment. Various treatment methods have been developed to remove NH_3-N, such as chemical precipitation[3], membrane technology [4] and adsorption [5]. Adsorption method is considered to be the most promising and effective approach due to its simplicity in operation and economically viable [6]. Malaysia generated huge amount of solid waste annually and agricultural waste contributed about 1.2 million tons per year [7]. The utilization of agricultural residue as low cost adsorbent has increased significantly over the past few years as these materials are abundant [8-10] and could reduce the solid waste disposal problem [11]. Nonetheless, the applicability of agricultural material for NH_3-N removal specifically has not received adequate interest as the availability of information is limited compared to other contaminants such as dye [12] and heavy metals [13]. Hence, this research work was intended to determine the adsorption performance of jackfruit seed adsorbent in batch and continuous fixed bed column processes with several experimental parameters. Jackfruit seed is an oblong cylindrical or rounded in shape fruit and composed of moisture (9.59 $\pm$ 0.1%), protein (0.09 $\pm$ 0.03%), lipid (0.03 $\pm$ 0.01%), ash (0.04 $\pm$ 0.04%), carbohydrates (93.64 $\pm$ 0.73%), amylose (25.54 $\pm$ 0.38%) and amylopectine (74.46 $\pm$ 0.38%) [14, 15].

2. Materials and methods

2.1 Materials

Jackfruit seeds were collected from market area located in Kota Belud, Sabah. The seeds were washed thoroughly with tap water and distilled water to remove surface adhered impurities before drying in an oven at 40°C for 48 hours. The seeds were then ground and sieved to a smaller size around 1-2 mm.

Ammonium chloride (NH_4Cl) was dissolved in 1 L of distilled water for adsorbate solution preparation and the ranges of adsorbate concentrations used in this study varied between 0 - 500 mg/L. 0.1 M of sodium hydroxide (NaOH) and sulphuric acid (H_2SO_4) were used for solution pH adjustment. Meanwhile, lignin (alkali) powder (Sigma-Aldrich, USA) and sodium chloride were used in determination of effect of lignin and ionic salt in adsorption performance respectively.

2.2 Batch adsorption experiment

A series of batch adsorption experiments were carried out at room temperature (29±1°C) for a period of time with constant adsorbent dosage of 20 g. The effect of ionic strength on the amount of NH_3-N adsorbed onto the adsorbent was evaluated at NaCl concentrations ranging from 0 to 2000 mg/L. Meanwhile, lignin was dissolved in distilled water to different concentration ranges (0-4000 mg/L) in order to assess the effect of lignin concentration on the adsorption performance. The experiment was held at constant volume of aqueous solution of 500 mL, 500 mg/L of initial concentration of NH_3-N, pH 7 and 20 g of adsorbent weight.

Sample solutions were withdrawn at certain time intervals (0-70 minutes) in order to determine the residual concentration at the time and then analyzed by using a Jasco UV-vis 650 Bio-spectroscope with a maximum wavelength of 425 nm following the Nessler method. The amount of NH_3-N adsorbed at equilibrium, Q_e (mg/g) and removal percentage (%) were calculated as follows [8]:

$$\text{Removal percentage (\%)} = [(C_o-C_e)/C_o]100 \quad (1)$$

$$\text{Adsorption capacity, } Q_e = [(C_o-C_e)V/M] \quad (2)$$

where C_o (mg/L) and C_e (mg/L) are respectively the initial and at time (t) ammonia nitrogen concentrations, V is the volume of aqueous solution (L) and M (g) is the mass of sample used as adsorbent. Each experimental parameter was repeated twice to avoid any discrepancy in experimental results and the results were taken as an average.

2.3 Fixed bed column studies

The dynamic behavior of a fixed bed column packed with jackfruit seed adsorbent in removing NH_3-N was evaluated by conducting continuous flow adsorption experiments in a fixed bed column of 6.5 cm internal diameter and 100 cm height. The adsorption column system was equipped along with a pump, flow meter and influent and effluent tank and operated from the upper boundary (down-flow mode) using a peristaltic pump (MasterFlex L/S Series Precision

Pump) and connected to a flow meter (TEFLON-PFA) as shown in Figure 1. The bottom of the bed column was covered neatly to avoid any loss of adsorbent. A thin layer of water is kept on the upper surface of adsorbent bed before the adsorbate solution was added into column in order to minimizing the channeling effect.

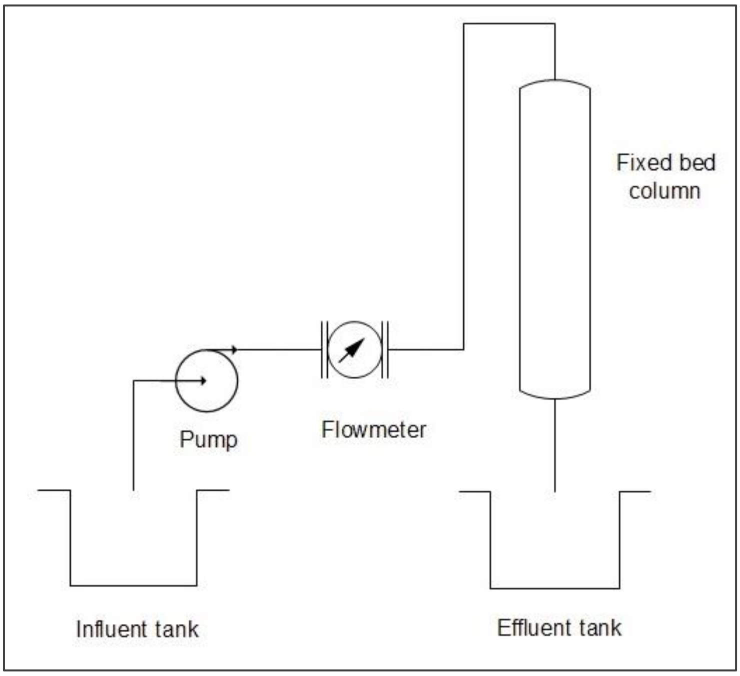

Fig. 1. Experimental setup for fixed bed adsorption column.

The breakthrough point is an effluent concentration (C_t) from the column that reaches about 0.1% of the influent concentration (C_o) whereas the exhaustion point is a point where the effluent concentration reaches 95%. Breakthrough curves were constructed as a function of each parameter studied (flow rate, ammonia nitrogen concentration and bed height) based on the ratio of influent and effluent concentration (C_o/C_t) at a given sampling time (t) [16, 17]. The effluent volume (V_{eff}), maximum bed capacity (Q_{total}), removal percentage (%) and equilibrium NH_3-N uptake ($Q_{eq(xp)}$) were calculated for each parameter. The effluent volume was calculated by using the following equation [18]:

$$V_{eff} = Qt_{total} \tag{3}$$

where V_{eff} is the effluent volume collected (L), Q is volumetric flow rate (mL/min), t_{total} is total flow time (min). The maximum bed capacity for a given flow rate and influent concentration is given as follows [16]:

$$q_{total} = QC_{ad}dt/1000 \quad (4)$$

where q_{total} is the maximum bed capacity (mg/g), C_{ad} is the concentration of adsorbed ammonia nitrogen (mg/L). The area under the curve of concentration of ammonia nitrogen adsorbed against time was used to determine the value of the integral. The total amount of ammonia nitrogen sent to the column is given by the following equation [17]:

$$M_{total} = C_o Q t_{total}/1000 \quad (5)$$

where M_{total} is total amount of ammonia nitrogen sent to the column (g), C_o is the initial concentration (mg/L), and t_{total} is the total flow time (min). Total removal percentage of ammonia nitrogen and equilibrium ammonium uptake can be measured by [17]:

$$\text{Removal Percentage (\%)} = (q_{total}/M_{total})100 \quad (6)$$

$$q_{eq(xp)} = q_{total}/x \quad (7)$$

where $q_{eq(xp)}$ is the equilibrium uptake (mg/g), and x is the amount of adsorbent (g).

2.4 Desorption

Desorption study was conducted by treating the ammonium-loaded adsorbent with various concentrations of H_2SO_4 and NaOH solutions in a series of batch experiments. The used adsorbent (20 g) was initially rinsed in distilled water and dried for 2 days at room temperature. The dried adsorbent then was agitated in the solutions for 70 minutes and the desorbed ammonia nitrogen concentration was determined by using UV-Vis spectrophotometer (Jasco UV-Vis 650 Bio-spectroscope) at maximum wavelength of 425 nm. All the experiments were repeated in duplicates and results are averaged.

3. Result and discussion

The present study is a continuation of the previous research work [19] which applied a screening process to 40 types of agricultural wastes in assessing the NH_3-N removal from aqueous solution. Jackfruit seed was selected as the best sorbent and used for further sorption studies due to the high adsorption capacity and its availability throughout the year. The effect of NaCl and lignin on ammonium adsorption was studied in this paper because it is important to evaluate the adsorbent performance at various solution conditions. For example, some of the potential solution for this adsorbent is drainage near the sea which is containing high salinity while palm oil mill effluent is high in lignin content.

3.1 Effect of sodium chloride concentration on adsorption of ammonia nitrogen

The influence of NaCl concentration on ammonium (NH_4^+) ion adsorption was studied at pH 7.0 with concentration ranges from 0 to 2000 mg/L. Figure 2 demonstrates that in the absence of salt, the NH_4^+ ions uptake showed the highest adsorption ability with adsorption capacity of 3.35 mg/g.

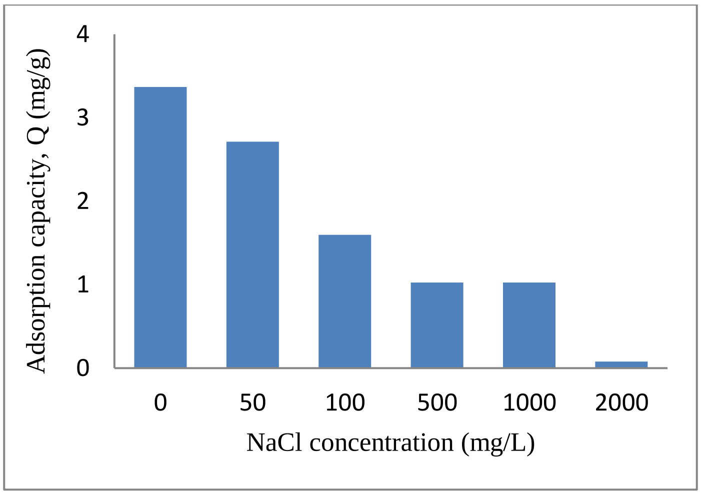

Fig. 2. Ammonia nitrogen removal by jackfruit seed with various NaCl concentration onto the adsorbent (contact time: 60 min, pH: 7.0, biosorbent weight: 20 g).

Further increase in the NaCl salt concentration exhibited a decreasing effect on the NH_4^+ ion uptake and this indicated that the cations from salt might subdue the positively charged NH_4^+ ion in the solution due to the competition between the cations present in salt and adsorbate species for the available negatively charged sites of jackfruit seeds adsorbent [20-22]. In this study, when the adsorbent is in contact with NH_4^+ ion species in aqueous solution, they are bound to be surrounded by an electrical double layer and an increase in ionic strength would influence the sorbent-sorbate interactions and therefore lower the adsorption performance [20, 23]. Previous studies showed similar results in lower adsorption capacity due to the increase in ionic salt concentrations [22, 24].

3.2 Effect of lignin concentration on adsorption of ammonia nitrogen

Lignin is a polymer of cross-linked phenyl propane units, conferring hydro-phobicity and provides structural rigidity and microbial resistance to plant cell walls [25, 26]. The adsorption capacity of ammonia nitrogen by jackfruit seed with different concentrations of lignin (0 to 4000 mg/L) was determined at a constant pH 7 and the experimental results are shown in Figure 3.

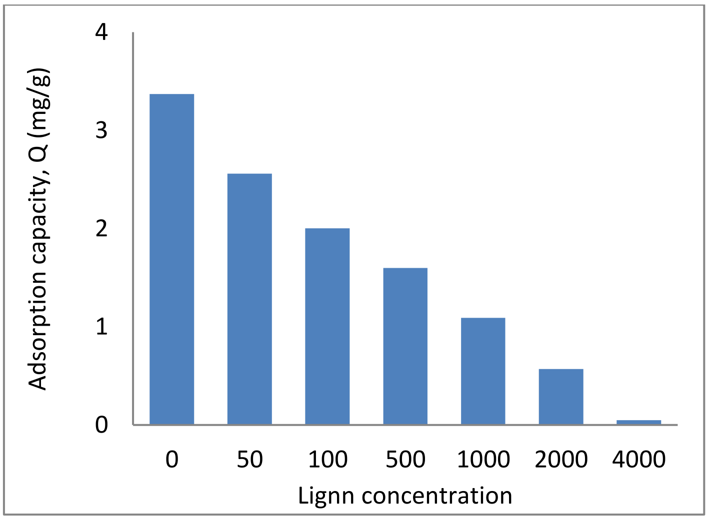

Fig. 3. Adsorption capacity of ammonia nitrogen by jackfruit seed adsorbent with various lignin concentration (Contact time: 60 min, pH: 7.0, Biosorbent weight: 20 g).

Maximum adsorption capacity of NH_3-N was achieved at 3.37 mg/g without any lignin addition and the sorption performances gradually decreased as the concentration of lignin increases up to 4000 mg/L. Due to the presence of hydrophobic lignin at the surface of jackfruit seed material, the strength and rigidity of the jackfruit seed cell walls are improved [26]. Besides, a plant with high lignin content may have some difficulties in breakdown process due to the intense bond formed between plant material and lignin itself. This eventually reduced the available surface area for the NH_4^+ ion to associate with and subsequently resulted in poor adsorption performance [27, 28].

3.3 Dynamic sorption of fixed bed adsorption column

The performance of the jackfruit seed as biosorbent in this study was evaluated based on the amount of ammonia nitrogen adsorbed (Q_{eq}), treated volume (V_{eff}), breakthrough point, and removal efficiency (%) at saturation condition. Table 1 tabulated all of the calculated parameters for each experimental breakthrough curves.

Table 1 Parameters of fixed bed column for ammonia nitrogen removal by jackfruit seed.

C_o (mg/L)	Z (cm)	Q (mL/min)	V_{eff} (mL)	m_{total} (mg)	q_{total} (mg)	q_{eq} (mg/g)	Removal percentage (%)
500	1	17	1445	722.50	77.31	3.86	10.70
500	5	17	2380	1190	373.89	4.51	31.42
500	10	17	3485	1742.50	807.48	4.86	46.34
500	10	28	4760	2380	715.90	4.31	30.08
500	10	43	6020	3010	558.66	3.36	18.56
500	10	57	5700	2850	366.80	2.21	12.87
100	10	17	4080	408	235.46	1.42	57.71
1000	10	17	2720	2720	984.39	5.93	36.19

*C_o= Initial concentration, Z= bed height, Q= flow rate, V_{eff}= volume effluent, q_{total}= maximum bed capacity, q_{eq}= equilibrium adsorbate uptake

3.3.1 Effect of flow rate

The effect of flow rate on adsorption of ammonia nitrogen by jackfruit seed adsorbent was determined by varying the inlet flow rates while the bed height and initial concentration was held constant at 10 cm and 500 mg/L respectively. The breakthrough curves for the adsorption process in a fixed bed column are presented in Figure 4.

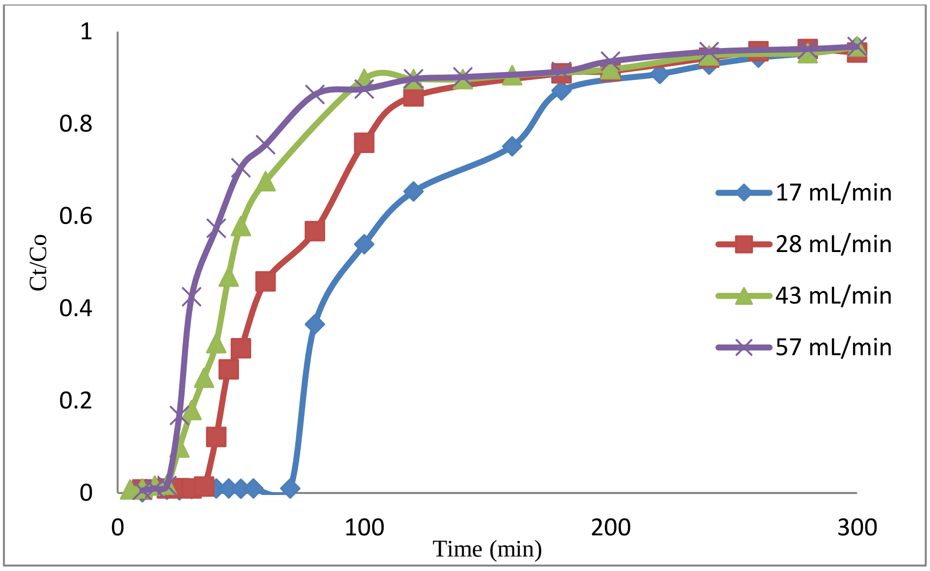

Fig. 4. Breakthrough curves of various inlet flow rate (Adsorbent bed height: 10 cm, initial influent solution concentration: 500 mg/L, inlet flow rates: 17, 28, 43 and 57 mL/min)

As indicated, the highest ammonia nitrogen removal was achieved at the lowest flow rate of 17 mL/min with 46.34% and adsorption capacity of 4.86 mg/g. Lower flow rate able to provide more contact time or residence time for the mass transfer diffusion process of the solute in the sorption bed [29]. Thus, prolonged bed saturation time can enhance the performances of adsorption process and increased the removal percentage of ammonia nitrogen. Moreover, the degree of axial flow dispersion in a column tends to increase along with the inlet flow rate as the Reynolds number increases due to the turbulence occurred [20]. Any fluid flow in column induces axial mixing dispersion which caused by the molecular diffusion and turbulent mixing around the particles. Theoretically, the degree of axial dispersion is somewhat greater at lower Reynolds number and this attributed to the effect of the greater hold-up of liquid in the laminar boundary layer surrounding particles which, combined with random fluctuations in the flow [21]. Greater axial dispersion in a column is undesirable as it reduces the efficiency of adsorption process. This result is in accordance to previous research done by [30]. It can be concluded that an increased in the flow rate reduced the removal efficiency as the insufficient residence time of mass transfer process to occur.

3.3.2 Effect of initial ammonia nitrogen concentration

Figure 5 depicts the effect of initial ammonia nitrogen solution concentration on the breakthrough curves with constant bed depth of 10 cm and inlet flow rate of 17 mL/min. The breakthrough time recorded for 100, 500 and 1000 mg/L were 75, 55 and 25 minutes respectively.

An increase in the initial adsorbate concentration significantly changed the trend of the breakthrough curves as it became steeper as the curves shifted to the left. A faster breakthrough and exhaustion time was achieved at higher ammonia nitrogen concentration and this resulted in reduction of bed sorption capacity obtained. This might be due to the greater concentration gradient at higher concentration and this provides higher driving force for the mass transfer process. Thus, the diffusion coefficient or mass transfer coefficient needs a shorter period for the transport process of NH_4^+ ion from the film layer to the surface of biosorbent [17, 29].

The maximum removal was observed at influent concentration of 100 mg/L, 10 cm of bed depth and 17 mL/min of flow rate. The detailed parameter values obtained from this experiment is tabulated in Table 1. These results proved that the adsorption process of ammonia nitrogen onto the biosorbent is highly concentration dependent which affects the saturation rate and breakthrough period [16]. A similar trend on the influence of influent concentration on adsorption process was observed in previous studies [17, 29].

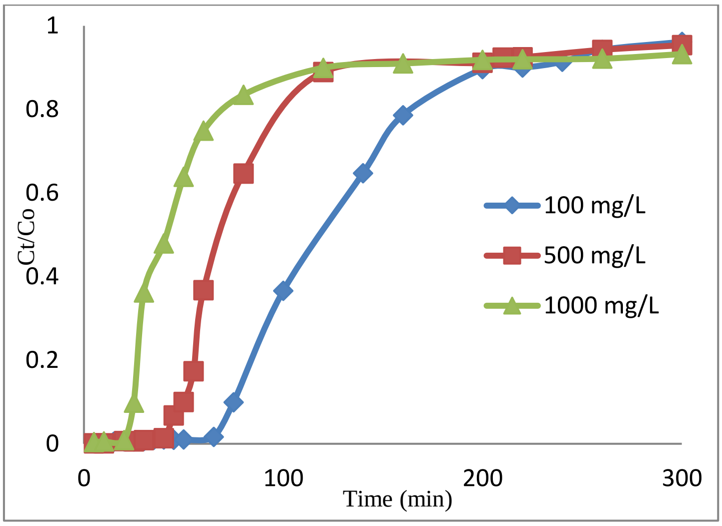

Fig. 5. Breakthrough curves of various initial adsorbate concentration ranges (Adsorbent bed height: 10 cm, inlet flow rate: 17 mL/min, adsorbate concentration: 100, 500 and 1000 mg/L).

3.3.3 Effect of different bed depths on breakthrough curve

The breakthrough curves showing ammonia nitrogen biosorption onto jackfruit seed adsorbent at different bed depths are presented in Figure 6. A constant influent concentration of 500 mg/L, pH 7.0 and flow rate 17 mL/min was passed through the column with various ranges of bed heights.

As can be seen from Figure 6, the breakthrough time increased remarkably from 15, 40 and eventually to 75 min with increasing bed height from 1 to 5 and 10 cm respectively. As the breakthrough time increased, the saturation time also showed an increasing pattern, which resulted in a broadened mass transfer zone. Thus, longer exhaustion time of the bed allows the NH_4^+ ions to come in contact with the binding sites of biosorbent [31]. Higher adsorbent bed offered more available binding sites for the attachment of ammonia nitrogen molecules and encouraged the diffusion mass transfer to dominate the adsorption process over the axial dispersion phenomenon [29]. Eventually, the volume of treated effluent, bed sorption and biosorption efficiency increased along with this as presented in Table 1. These results indicated that a higher biosorbent bed height is desirable for better adsorption column performance as reported by [18, 31].

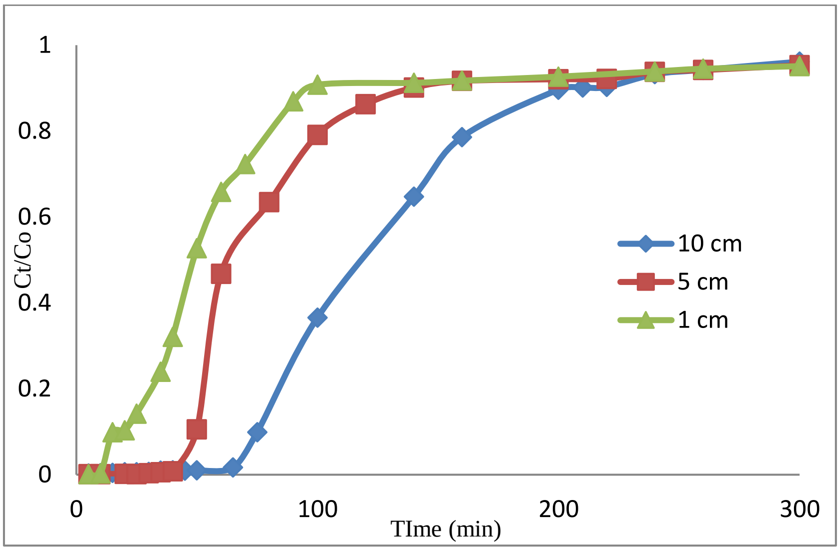

Fig. 6. Breakthrough curve of different bed height (Initial influent concentration: 500 mg/L, inlet flow rate: 17 mL/min, biosorbent bed depth: 1, 5 and 10 cm).

3.4 Desorption

Figure 7 demonstrates the desorption performance of NH_4^+ loaded adsorbent at different pH values. The maximum desorbed amount of NH_4^+ ions from the used adsorbent was obtained at lowest pH analyzed (pH 3) with a released amount of 0.42 mg/g. At acidic medium, the NH_4^+ ion replaced by hydrogen (H^+) ions at the surface of adsorbent thus resulting in the release of NH_4^+ ions to the solution, However, the desorbed amount value then dropped significantly as the pH value increased up to neutral pH 7 and hit the lowest desorption amount with 0.09 mg/g. As the pH moves towards the strong alkaline solution, the graph shows that the amount of NH_4^+ ions desorbed increased once again and reached a desorption capacity of 0.36 mg/g at pH 11. This is likely due to the hydroxide (OH^-) ions would react with weak ionized NH_4^+ that was adsorbed onto the surface of adsorbent in higher pH solution. The interaction can be written as below:

$$NH_4^+ (aq) + OH^- (aq) \rightarrow NH_3 (v) + H_2O (l) \quad (8)$$

It can be concluded that only a small amount of desorbed amount was able to be recovered from jackfruit seed adsorbent when reacted with 0.1 M of NaOH and H_2SO_4. Nevertheless, desorption could be regarded as solely surface reaction and the attachment between the NH_4^+ ions electrostatic attraction is weak as it could be desorbed under neutral pH water [32]. This desorption result is in accordance with previous studies on desorption of NH_4^+ ions using hydrogel, as the highest amount of desorption was observed at pH 2 and 12 [32].

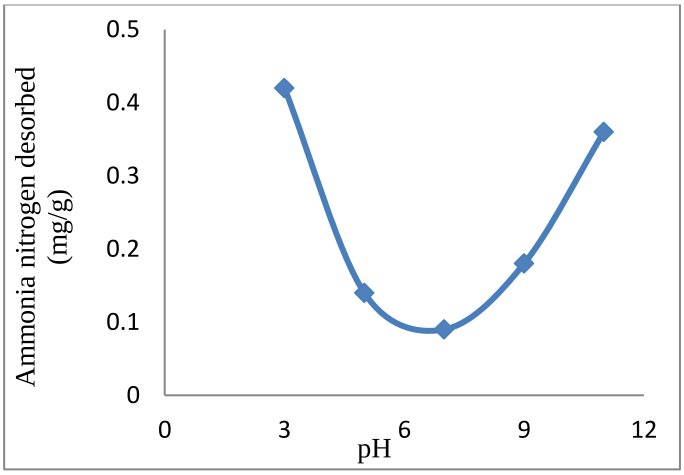

Fig. 7. Effect of pH on desorption of ammonia nitrogen from jackfruit seed adsorbent (initial ammonia nitrogen concentration: 500 mg/L, contact time: 60 min, biosorbent weight: 20 g).

4. Conclusion

This study evaluated the ability of jackfruit seed adsorbent for adsorption of NH_3-N under a batch and fixed bed continuous studies. The results depicted that NH_3-N uptake by jackfruit seed adsorbent decreased significantly upon the addition of NaCl and lignin into the solution. As the ionic salt concentration increased, the cations present in the salt was likely defeat the NH_4^+ ions to bind to negatively charged surface adsorbent thus lower the adsorption capacity.

The NH_4^+ ions uptake decreased from 3.37 mg/g as the lignin concentration increased due to better resistance and rigidity provided by lignin to the jackfruit seed cell wall. Meanwhile, the breakthrough curves obtained from the continuous column experiments followed the S-shaped characteristic with maximum removal rate was achieved at the lowest flow rate of 17 mL/min, low initial concentration of 100 mg/L and highest bed depth of 10 cm. Maximum desorbed amount of NH_3-N was obtained at pH 3 with desorption capacity of 0.42 mg/g. Hence, it can be concluded that jackfruit seed is potentially to be used as an alternative adsorbent for NH_3-N removal from aqueous solution.

Acknowledgements

This study was financially supported by Ministry of Higher Education Malaysia and Universiti Malaysia Sabah under research Project Grant FRG0420-TK-1/2015

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