

# The efficiency of modified powdered activated carbon for removal of ammonia nitrogen from aqueous solution: a process optimization using RSM (Response Surface Methodology), adsorption isotherm and kinetic study

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## ABSTRACT

The objective of this study was to determine the performance of modified commercial powdered activated carbon (MCPAC) in removal of ammonia from aqueous solution. The effects of adsorbent dosage (0.5–1.5 g/L), ammonia concentration (100–200 mg/L), pH (3 to 9) and contact time (2 to 120 min) were examined. In this study, experiments were performed based on Response Surface Methodology (RSM). The adsorption isotherm was evaluated using Freundlich and Langmuir models. Kinetics study was analyzed using pseudo first order, pseudo second order and particle penetration kinetic models. By increasing adsorbent dosage, pH and contact time, the removal efficiency increased. According to CCD results, the MCPAC was fitted to a quadratic equation. Also, ammonia adsorption for MCPAC followed the Langmuir ( $R^2 = 0.9831$ ) isotherm model. The maximum adsorption capacity for MCPAC was 40.323 mg/g. The analysis of adsorption kinetic for MCPAC indicated that ammonia adsorption was well fitted by the pseudo second order kinetic model. As thus, we concluded that MCPAC could be considered as an appropriate and economical alternative adsorbent for water and wastewater treatment due to its high ammonia adsorption capacity.

**Keywords:** modified activated carbon; ammonia; RSM; aqueous solution; adsorption isotherm

## Introduction

Nowadays, industrial and agricultural activities have been increasing, which subsequently leads to discharge of various types of organic and inorganic contaminants into the environment and receiving water. Accordingly, conserving the environment and public health in societies has become a notable challenge.<sup>1</sup>

Ammonia can have negative impacts on aquatic ecosystems and on public health of

societies.<sup>2</sup> Considerable amounts of ammonia could get into streams as a result of activities like those of the steel industry, fertilizers, refineries, food production factories and glass industries, and are estimated at approximately 45 million tons per year.<sup>3,4</sup> Thus, elimination or reduction of ammonia in the receiving environment is necessary. Conventional wastewater treatment plants cannot afford environmental discharge standards. Particularly when notable amounts of wastewater have been discharged into receiving waters, it turns out to be a substantial concern.<sup>1</sup> So far, various methods for ammonia removal have been applied such as chemical oxidation, ultrasound, air stripping, breakpoint chlorination, nitrification and denitrification, ion exchange and adsorption.<sup>5-7</sup> Adsorption on activated

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carbon has been known to be an effective technology for removal of organic and inorganic contaminants. Recently, application of modified activated carbon has been increased, and satisfactory results have been obtained.<sup>8</sup> Adsorption process has been increasingly employed compared to other techniques due to its characteristics such as design flexibility, simple operation and low cost.<sup>9,10</sup> Several methods have been studied by different chemical agents to develop surface-modified activated carbon to add functional groups and porosity on modified activated carbon which, compared to the unmodified type, has been improved.<sup>10</sup> In a study by Marcano et al. entitled "Improved Synthesis of Graphene Oxide," the results showed that modified graphene has high efficiency in removing contaminants. Marcano et al. applied the agents sulfuric acid/ sodium nitrate/ sodium permanganate simultaneously to improve and reinforce graphite electrodes (adding functional groups). This small change in the procedure made the production of highly oxidized GO in a single reaction vessel significantly more practical, compared to other methods.<sup>9</sup> So far, these three chemicals have not been used simultaneously to modify the activated carbon, and the removal of ammonia nitrogen by the above-mentioned modified activated carbon has not yet been investigated. Therefore, chemical agents such as sulfuric acid, sodium nitrate and potassium permanganate were used simultaneously to improve powdered activated carbon and to remove ammonia nitrogen from aqueous environments.

In the present study, RSM was applied to analyze and optimize the effect of different variables, including adsorbent dosage, contact time, adsorbate concentration and pH, on ammonia nitrogen removal from aqueous solution. Process optimization by RSM demonstrated the effect of operating variables and also the interactive effects of independent variables on response. Adsorption isotherm was evaluated using Langmuir and Freundlich isotherm models and kinetic study was performed to analyze the adsorption kinetics model.

## Materials and Methods

### *ARMA multivariate models*

This experimental study was conducted in batch mode and the potential of modified commercial powdered activated carbon (MCPAC) with H<sub>2</sub>SO<sub>4</sub>, NaNO<sub>3</sub> and KMnO<sub>4</sub> for removal of ammonia nitrogen from aqueous solution was studied in a pilot scale.

All chemicals and reagents were purchased with a purity of 99.99% from Merck Co., Germany. It should be noted that the deionized water which was procured in a laboratory was used to prepare the desired standard solutions. The commercial powdered activated carbon was purchased from Merck Co., Germany, with code 102183.

### *Preparation of MCPAC*

At first, 5 g of powdered activated carbon was weighted and 69 ml of H<sub>2</sub>SO<sub>4</sub> and 1.5 g of NaNO<sub>3</sub> were added to solution. Then, 9 g of KMnO<sub>4</sub> was added to solution slowly and volume was increased to 500 cc. Temperature was kept below 20 °C in this step. Then, the solution was heated to 35 °C and was stirred on magnetic shaker for 7 h. Afterward the solution was cooled to room temperature and furthermore, 400 ml of ice and 3 ml of hydrogen peroxide (30%) were added into the prepared solution. The solution was centrifuged at 4,000 rpm for 4 h. Finally, the supernatant was taken from the solution slowly and remaining solid was washed with 200 ml of distilled water, 200 ml of 30% hydrochloric acid and 200 ml of ethanol. Then, the samples were dried and the prepared solution was sieved (80 mesh). It must be noted that every stage of sampling, storage and the experimentation was carried out based on standard method for water and wastewater examination.<sup>11</sup>

### *Determination of adsorbent's characteristics*

The characteristics of MCPAC such as surface morphology, surface functional group, chemical characteristics and zero point charge were determined through laboratory diagnostic techniques using scanning electron microscopy (SEM), Fourier transform infrared (FTIR)

transmission spectra, X-ray fluorescence (XRF), and  $\text{pH}_{\text{zpc}}$ .

### The system operation

This study was performed at pH: 3, 6, 9 for determination of optimum pH of adsorbent. In order to determine the effect of contact time and pH on ammonia nitrogen removal by MCPAC, the adsorbent dosage of 1.5 g, constant concentration of ammonia nitrogen of 150 mg/L, and contact times of 5, 10, 20, 30, 60, 90 and 120 min were examined. In the next step, in order to determine the effect of adsorbent dosage, the different doses of MCPAC (0.5–1.5 g) were analyzed for removal of initial ammonia nitrogen concentration of 150 mg/L. It should be considered that in alkaline pH a small part of ammonia nitrogen evaporates as  $\text{NH}_3$ . Therefore, to achieve the amount of pure adsorption, the control samples were analyzed by different concentration of 50, 100 and 200 mg/L and pH: 9 to distinguish the amount of volatile ammonia from the adsorbate amount.

### Experimental design and mathematical modeling

In this study, the experiments were designed based on the central composite design (CCD) and analyzed by response surface methodology (RSM) which provided proper statistical tools to design and analyze experiments for process optimization.

A model in the form of Eq. (1) was presented in order to fit the experimental data by optimization. The relationship between response, input and quadratic equation model to predict the optimal variables was identified through the following equation:

$$Y = \beta_0 + \beta_i X_i + \beta_j X_j + \beta_{ii} X_i^2 + \beta_{jj} X_j^2 + \beta_{ij} X_i X_j + \dots \quad (1)$$

where Y, i, j,  $\beta$ , X are process response, linear coefficient, quadratic coefficient, regression coefficient and coded independent variables, respectively.<sup>12</sup> In the present work, CCD was applied to study three different factors, i.e., initial ammonia nitrogen concentration, adsorbent dosage and solution pH. Accordingly, Twenty experiment runs were designed. The

designs consisted of five levels (–0.5, –1, 0, +1, +0.5) with five repetition points in the surface (Table 1). The obtained results, based on CCD, were analyzed by analysis of variance (ANOVA). The results can be demonstrated by 3-Dimensional plots with regard to simultaneous effect of independent variables on the responses as well.

Table 1. Experimental condition designed by Design Expert software

Run	Initial concentration	Adsorbent dosage (g)	pH
1	200	1.5	3
2	200	1.5	9
3	150	0.75	6
4	150	1	6
5	100	0.5	3
6	150	1	7.5
7	175	1	6
8	100	0.5	9
9	200	0.5	9
10	200	0.5	3
11	150	1	6
12	150	1	6
13	150	1.25	6
14	100	1.5	3
15	100	1.5	9
16	150	1	6
17	150	1	6
18	150	1	4.5
19	150	1	6
20	125	1	6

### Determination of adsorption isotherm

Adsorption isotherm of modified activated carbon at eight initial ammonia concentrations of 50–300 mg/L and contact time of 2–120 min were fitted to Langmuir and Freundlich isotherm models. Accordingly, the adsorption of activated carbon was compared by presented models and analyzed by the adsorbent efficiency.<sup>13</sup>

### Determination of kinetic reaction

In order to determine the reaction kinetics, experiments were performed at different contact times of 2–120 min with other constant variables (pH: 9 and adsorbent dosage of 1.5 g). The obtained results were fitted to pseudo first order, pseudo second order and particle penetration kinetic model.<sup>14</sup> The constant rate of

adsorbant components was determined and the adsorption rate using modified activated carbon was analyzed.

## Results and Discussion

### FTIR, SEM and XRF analysis

The obtained results of FTIR spectra showed that the surface of MCPAC had

different functional groups. The peaks showed in Fig. 1 for MCPAC are related to C-O groups, carbonyl, CO<sub>2</sub>, hydroxyl and carboxyl.<sup>15</sup> According to FTIR spectra, the modified activated carbon showed the lower transmission which reflected that MCPAC was more capable for adsorption.<sup>16</sup>

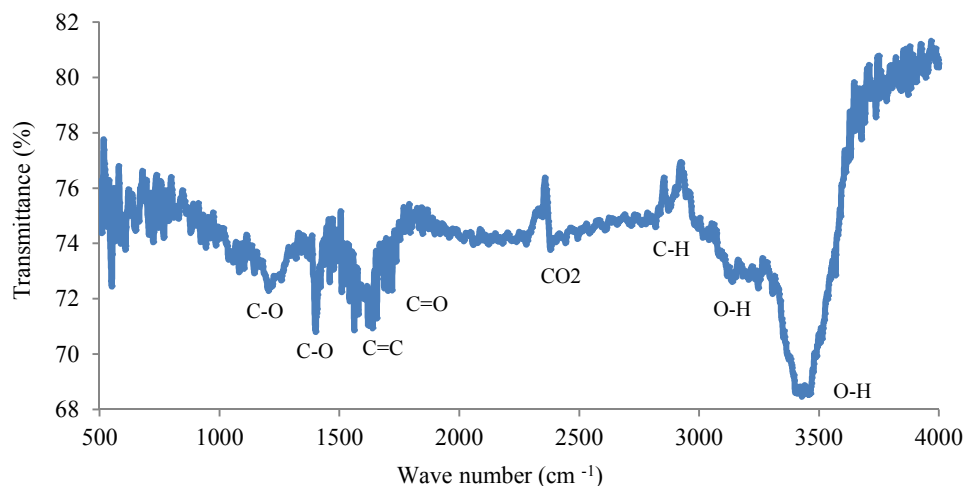


Fig. 1. FTIR spectra of MCPAC

SEM images were employed aiming to observe the surface physical morphology of the adsorbent with magnification of 600, 1,500 and 3,000 (Fig. 2). As is demonstrated, based on the SEM images, noticeable changes appeared in the number of pores at adsorbent surface after modification of powdered commercial activated carbon.

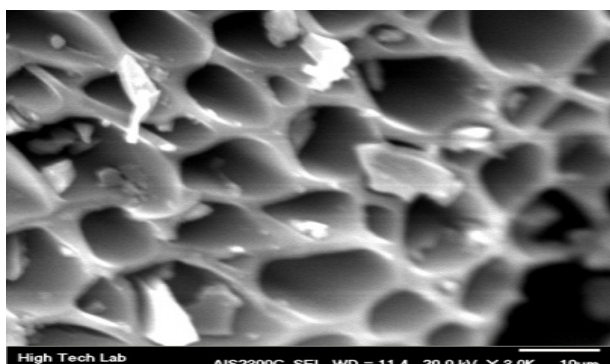


Fig. 2. SEM micrographs (3000X) of MCPAC

Table 2 shows XRF analysis which was performed for MCPAC in order to determine the

chemical compound of adsorbent. The obtained results indicate that the adsorbent, in addition to main composition, has different amounts of other chemical compounds.

Table 2. XRF analysis the MCPAC

Compound	Element	MCPAC (%)
Loss on ignition	Carbon	83.02
MgO	Magnesium	0.921
Al <sub>2</sub> O <sub>3</sub>	Aluminum	-
SiO <sub>2</sub>	Silicon	0.9767
P <sub>2</sub> O <sub>5</sub>	Phosphorus	-
SO <sub>3</sub>	Sulfur	13.66
Cl	Chlorine	0.1257
K <sub>2</sub> O	Potassium	1.033
CaO	Calcium	0.1201
TiO <sub>2</sub>	Titanium	0.014
MnO	Manganese	0.04417
Fe <sub>2</sub> O <sub>3</sub>	Iron	0.061

### Determination of pH<sub>ZPC</sub>

The results of the experiment show that pH<sub>ZPC</sub> was 5.6 for MCPAC. The results of pH<sub>ZPC</sub> are shown in Fig. 3.

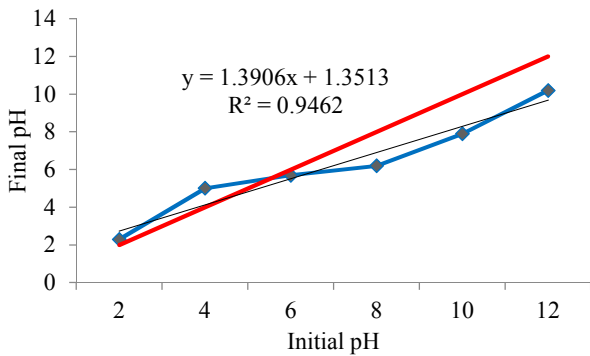


Fig. 3. The initial solution pH against the final pH in order to determination the  $pH_{zpc}$  MCPAC

**Statistical analysis**

According to CCD, the different factors such as adsorbent dosage (A), adsorbate dosage (B), and solution pH (C) were coded. The results

show that maximum ammonia nitrogen removal efficiency by MCPAC was accomplished at 97.08%. The maximum adsorption capacity of ammonia nitrogen by MCPAC reached 79.04 mg/g. The result of model prediction was 79.8 mg/g, which verified the validity and reliability of analysis. In the present study, the degree polynomial models was used for data fitting. The results of variance analysis show that ammonia nitrogen removal efficiency by MCPAC was fitted to quadratic equation.

Also, regression equation, proper model for ammonia nitrogen removal, correlation coefficient ( $R^2$ ), reliability and validity, standard deviation and *P*-value are presented in Table 3.

Table 3. ANOVA results for the equation of the Design Expert for MCPAC

Modified equation with significant terms	Type of model	$R^2$	Adeq. precision	Standard deviation	<i>P</i> -value
$91.55+3.43A-5.154B+2.545C-5.101B^2+0.357AB+0.587AC+0.58BC$	Quadratic	0.997	91.299	0.38	<0.05

**Effect of adsorbent dosage and adsorbate concentration at different pH**

Figure 4 shows the simultaneous effect of adsorbent dosage and adsorbate concentration on ammonia nitrogen removal efficiency at different pH (3-9) using MCPAC. The results

show that by increase of adsorbent dosage and reduction of adsorbate concentration, ammonia nitrogen removal increased. Also, based on Figs. 4 and 5, ammonia removal increased by increasing pH from 3-9.

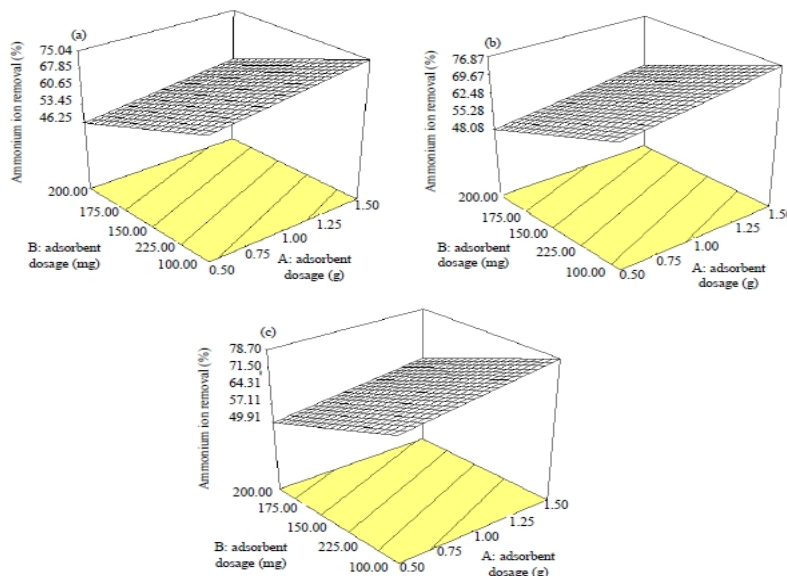


Fig. 4. Response surface plot for ammonia nitrogen removal by MCPAC at different pH: a) pH= 3, b) pH= 6 and c) pH=9

Figure 5 shows the simultaneous effect of adsorbent dosage and adsorbate concentration

on adsorption capacity of ammonia nitrogen by MCPAC at different pH of 3-9. It was observed



that by reducing the adsorbent dosage and increasing the adsorbate concentration, the

adsorption capacity of ammonia nitrogen was increased.

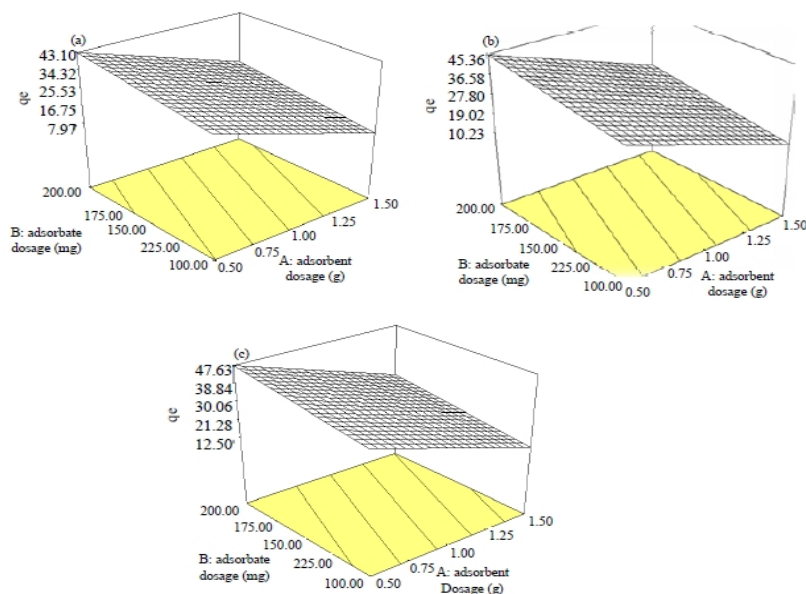


Fig. 5. Response surface plot for adsorption capacity of ammonia nitrogen by MCPAC at different pH: a) pH= 3, b) pH= 6 and c) pH=9

### Effect of solution pH

The simultaneous effect of pH (3, 6 and 9) at different contact times (from 2–120 min), constant concentration of 200 mg/L, and adsorbent dosage of 1.5 g for MCPAC are demonstrated. As can be seen in Fig. 6, the removal efficiency increased when pH increased from 3–9. Therefore, pH: 9 was determined as optimum pH. Our investigation showed that ammonia removal efficiency increased by increase of solution pH. It was related to adsorbent surface that had negative charge at a higher pH than  $pH_{zpc}$ , with regard to the fact that ammonia has been categorized in cationic compounds which create the electrostatic attraction force between adsorbent and targeted contaminant. So, as a result, the removal efficiency was increased. Lesser pH than  $pH_{zpc}$  led to increase of the positive charge at adsorbent surface which created the repulsive force between adsorbent and the cationic contaminant which subsequently reduced the ammonia removal efficiency. In the present study, the isoelectric point ( $pH_{zpc}$ ) for MCPAC was 5.6. This finding is consistent with findings of the study by Zhu et al., who investigated the

removal of  $NH_4^+$  D-N from aqueous solution by using activated carbon derived from rice husk.<sup>3</sup> Zheng et al. studied rapid and wide pH-independent ammonium removal using a composite hydrogel with three-dimensional networks and maximum adsorption capacity was observed at pH 4–8.<sup>15</sup> Effect of  $Na^+$ -impregnated activated carbon on the adsorption of  $NH_4^+$ -N from aqueous solution was studied by Shi et al., and their results showed that the optimum  $NH_4^+$ -N removal efficiency was accomplished at neutral pH.<sup>2</sup>

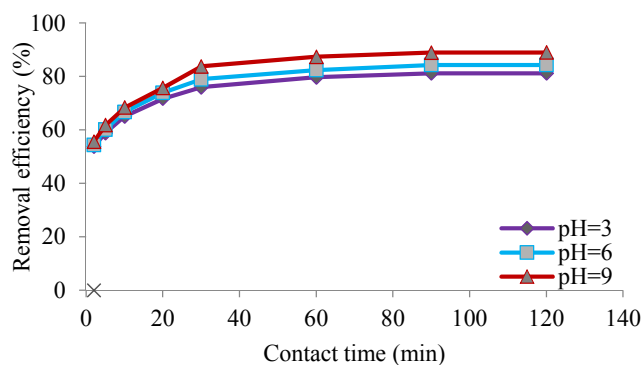


Fig. 6. Effect of pH on efficiency of ammonia nitrogen removal MCPAC

### **Effect of adsorbent dosage**

The obtained results of variant adsorbent dosages on efficiency of ammonia removal by MCPAC demonstrated that nitrogen removal efficiency was increased when the adsorbent dosage increased for MCPAC (Fig. 7). Also, adsorbent dosage of 1.5 g was determined as optimum adsorbent dosage for MCPAC. It has also been found that by increasing the adsorbent dosage, the adsorption capacity decreases, which leads to increase in the efficiency of ammonia removal by MCPAC. By increasing the adsorbent dosage from 0.5–1.5 g/L, adsorption capacity decreased from 79.024 to 30.219 for MCPAC. The increment of adsorbent dosages reflected on the increment of adsorption surface which enhanced the access of contaminant molecules to adsorbate sites over the adsorbent surface that caused the increase of removal efficiency. On the other hand, increasing the adsorbent dosage means faster adsorption of contaminant from solution and decreases the contaminant concentration in the solution. This causes the reduction of available molecules to create the maximum surface coverage that then leads to reduction of the adsorbate contaminant per unit weight of adsorbent. This trend demonstrates that the adsorbate particles over the adsorbent surface with remaining molecules in the solution lead to closing of adsorbent internal pores or cause the accumulation and integration of adsorbent particles, which subsequently leads to decrease in the active sites for adsorption.<sup>17</sup> It was also found that the increase in ammonia removal efficiency was remarkably faster when the adsorbent dosage increased from 0.5–1 g/L, and by increasing the adsorbent dosage from 1–1.5 g/L, the increase of removal efficiency continued with a slower trend.

This means that adsorption occurs faster in adsorbent surfaces at higher adsorbent dosages which leads to decrease of adsorbate in solution compared to lower adsorbent dosages.<sup>4</sup> These findings are identical to the results of the study by Zhu et al. for the removal of  $\text{NH}_4$  D-N from aqueous solution by using the activated carbons derived from rice husk. The results of Zhu et al.

showed that the removal efficiency of ammonia nitrogen increases significantly by increasing the adsorbent dosage from 0.2–2 g, which reflected that removal efficiency increases from 10.12% to 48.12%.<sup>3</sup> Also, the results of Hussain et al. on the physico-chemical method for ammonia removal from synthetic wastewater using limestone and GAC in batch and column studies showed that increasing the adsorbent dosage is related to the increment of contact time and, as a result, the removal efficiency increases.<sup>18</sup>

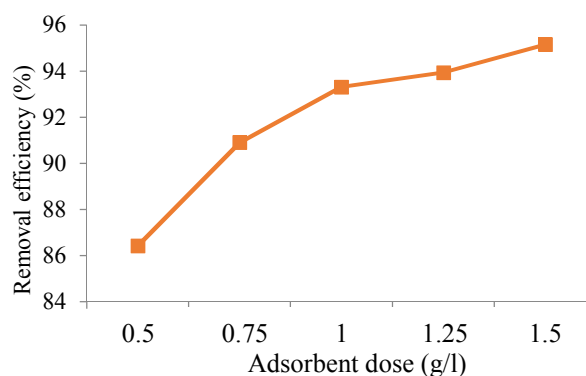


Fig. 7. Effect of adsorbent dosage on efficiency of ammonia nitrogen removal (initial ammonia nitrogen concentration of 150 mg/L, contact time of 120 min and pH=9)

### **Effect of ammonia nitrogen concentration**

Figure 8 shows the effect of initial ammonia nitrogen concentration on removal efficiency by MCPAC. As can be seen by increasing the ammonia nitrogen concentration from 50 to 300 mg/L, the removal efficiency was then decreased. The maximum removal efficiency was accomplished to 100% by MCPAC. Also, investigation showed that there is little difference in removal efficiency by MCPAC at low concentrations, while as the initial concentration increased, the difference in removal efficiency increased.

The results found that removal efficiency decreased from 90.94% to 52.69% by MCPAC when ammonia nitrogen concentration increased from 50–300 mg/L at constant conditions of other variables, which reflects the fact that adsorption is strongly dependent upon initial concentrations of the solution. The adsorption capacity increased by increasing the

contaminant concentration.<sup>19</sup> Because the adsorbent has specific and limited adsorption sites, more adsorption sites are available at low concentration and subsequently faster adsorption occurred and led to increase of removal efficiency. But, at higher concentrations, by increase of the adsorbate on the adsorbent surface, the adsorption sites over the surface were saturated and, as a result, they reduced the removal efficiency. Adsorption capacity increased by increasing the initial ammonia nitrogen concentration, which may be attributed to interaction between the adsorbent and adsorbate contaminant.<sup>19,20</sup> Lebedynets et al. studied the adsorption of ammonium ions onto a natural zeolite and their results revealed that the adsorbate ammonium ion increased by decreasing the initial concentration and adsorbate volume as well.<sup>21</sup>

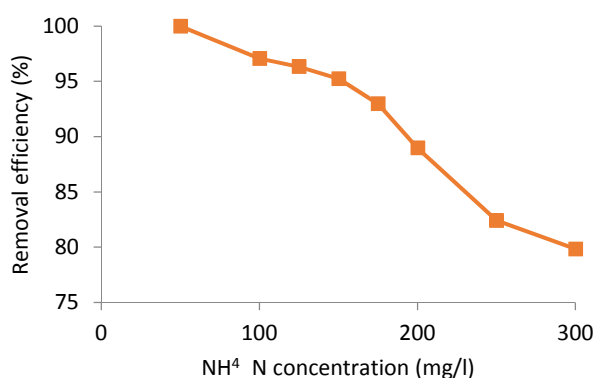


Fig. 8. Effect of initial ammonianitrogen concentration on removal efficiency (adsorbent dosage of 1.5 g, contact time of 120 min and pH= 9)

### Effect of contact time

Figure 9 shows the effect of contact time on ammonia nitrogen removal efficiency by MCPAC. The results show that by increasing contact time from 2–120 min at different ammonia concentrations, the removal efficiency increased. At beginning of adsorption reactions, due to numerous adsorption sites and notable difference between concentrations of adsorbate material in the solution and the amount of adsorbate material over the adsorbent surface, the removal efficiency increased.<sup>16</sup> Over time, the increasing trend of ammonia removal has continued by moderate slope which may be related to a contaminant layer over the surface. Also, over time, it was becoming difficult to occupy the remaining surface sites because there was a type of repulsion between adsorbed molecules and soluble molecules. The obtained results show that in the first 60 min, more than 80% of ammonia nitrogen was removed by modified adsorbent. Based on experimental results, the adsorption was not much changed after 120 min. In this situation, the adsorbate ammonia nitrogen over the surface was equal to amount of ammonia nitrogen in aqueous solution. So, adsorption equilibrium time was attained in 120 min of contact time. Results of the study of Zabochnicka et al.

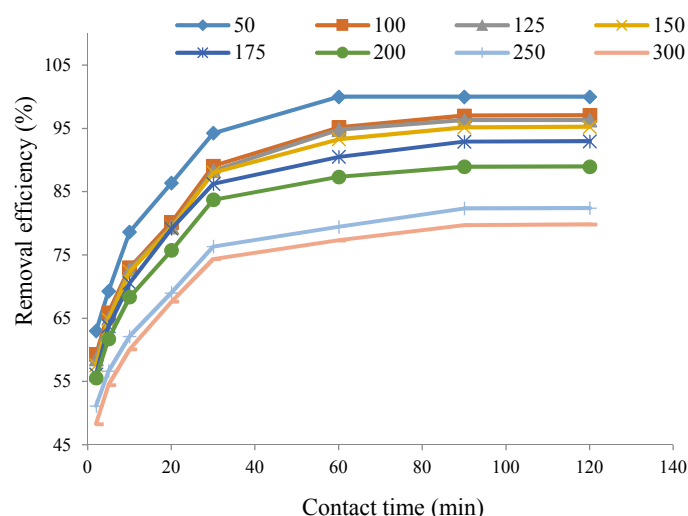


Fig. 9. Effect of contact time on efficiency of ammonia nitrogen removal (initial ammonia nitrogen concentration of 50-300 mg/L, adsorbent dosage of 1.5 g and pH=9)



regarding the removal of ammonia by clinoptilolite show that by increasing contact time the removal efficiency improved and the adsorption equilibrium was determined at 180 min.<sup>22</sup> Lebedynets et al. studied the adsorption of ammonium ions onto a natural zeolite and found that the removal efficiency did not change after 24 h.<sup>21</sup> Contradictions in the findings of the present work with other researches may also be related to structural differences of adsorbent and chemical compounds. But most publications about ammonia removal using various adsorbents have admitted that removal of ammonia nitrogen increases when contact time increases.

### Adsorption isotherms

The regression coefficient values were determined for ammonia nitrogen adsorption using the MCPAC by the Langmuir and

Freundlich isotherm models (Table 4). The value of regression coefficient showed that MCPAC followed the Langmuir ( $R^2 = 0.98$ ) isotherm model.

Table 4. Parameter value of Langmuir and Freundlich isotherm model for MCPAC

Freundlich			Langmuir		
$R^2$	n	$k_f$	$R^2$	b	$q_e$ (mg/g)
0.9764	4.55	15.019	0.9831	0.22	40.323

### Adsorption kinetics

The correlation coefficient values based on different kinetic models (pseudo first order, pseudo second order and particle penetration) demonstrated that the ammonia nitrogen removal by MCPAC followed the second order adsorption kinetic model. The calculated parameters from the kinetic models are presented in Table 5.

Table 5. The calculated parameters of pseudo-first order, pseudo-second order and particle penetration according to adsorbent type for MCPAC

Particle penetration		Pseudo-second order			Pseudo-first order		
$R^2$	$k_p$	$R^2$	$q_e$ (mg/g)	$K_2$	$R^2$	$q_e$ (mg/g)	$K_1$
0.8599	1.1664	0.9997	30.40	0.0124	0.8543	9.23	0.042

The obtained results from the linear regression coefficient for pseudo first order, pseudo second order and particle penetration for MCPAC found that adsorption kinetic followed the pseudo second order kinetic model.

This means that the adsorption process depends on the adsorbate concentration, because the pseudo second order kinetic model has been established based on adsorption capacity.<sup>23</sup> So, it can be concluded that most of the adsorption in MCPAC takes place through chemical adsorption on the layer over the surface. The results of the present study are consistent with the results of studies from Arslan<sup>24</sup>, Zhu<sup>3</sup> and also Zheng.<sup>25</sup>

### Conclusion

The results of this study reveal that ammonia nitrogen removal efficiency is more satisfying at alkaline pH for MCPAC. It was observed that the adsorption equilibrium was attained with 120 min of contact time. Also, efficiency of ammonia nitrogen removal

increased by increasing adsorbent dosage and reducing initial ammonia nitrogen concentration. The adsorption isotherm and kinetic study of ammonia nitrogen showed that MCPAC followed the Langmuir isotherm. The kinetic study of adsorbent followed the pseudo second order kinetic model. Also, the results of the central composite design showed that MCPAC was fitted to the quadratic equation, and that the prediction of the model and the experimental findings were in close agreement. It was concluded that MCPAC can be considered as an appropriate and economical alternative for water and wastewater treatment due to the high adsorption capacity of MCPAC.

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