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Removal of nitrate—nitrogen from aqueous medium by adsorbents derived from pomegranate rind

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ABSTRACT

Thermally treated carbons derived from pomegranate rind were investigated to find the suitability of its application for the removal of nitrate in aqueous solution through adsorption process. Two types of activation namely thermal at 200, 300, and 400°C and boiling treatment (boiling at 150°C) were used for the production of the adsorbents. A control (untreated pomegranate rind (UPR)) was used to compare the adsorption capacity of the adsorbents produced from these processes. The results indicated that the thermally treated carbon derived at the temperature of 400°C showed maximum adsorption capacity in the aqueous solution of nitrate. Batch adsorption studies showed an equilibrium time of 6 h for the thermally treated carbon derived at 400°C. It was observed that the adsorption capacity was higher at lower pH (2–3) and higher value of initial concentration of nitrate (200 mg/L). The equilibrium data fitted better with the Freundlich adsorption isotherm compared to the Langmuir. Kinetic studies of nitrate adsorption onto adsorbents were also studied to evaluate the adsorption rate.

Keywords: Adsorbents; Adsorption; Pomegranate rinds; Thermal treatment; Physical; Treatment; Nitrate

1. Introduction

Nitrate contamination in surface and ground water has become an increasingly important problem for all over the world. Although nitrate is found in moderate concentrations in most of the natural waters, higher levels in ground water mainly result from human and animal waste, and excessive use of chemical fertilizers. The other most common sources of nitrate are uncontrolled land discharges of municipal and industrial waste waters, overflowing septic tanks, processed food, dairy and meat products, and decomposition of decaying organic matters buried in the ground. These fertilizers and wastes are sources of nitrogen containing compounds, which are converted to nitrates in the soil. Nitrates are extremely soluble in water and can move easily through soil into the drinking water supply. US Environmental Protection Agency (EPA) has set the maximum contamination level as 10 mg/L of NO_3^--N[1,2].

The high concentration of nitrate in drinking water leads to the formation of nitrosoamine, which is related to cancer and increases the risk of diseases such as methanoglobinemia in newborn infants [3]. Several methods that serve to reduce nitrate in drinking water have been presented [4–7]. The use of biological reactor seems to be the most promising

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technique in the treatment of high nitrate concentration. However, maintaining biological processes at their optimum conditions is difficult, and the problem of contamination by dead bacteria has to be solved to make such processes safety enough to utilize in drinking water treatment. On the other hand, adsorption is a very feasible process for *in situ* treatment of underground and surface water, primarily due to its ease of application.

An increasing number of bioresources has been utilized in the preparation of adsorbents, and some studies show that certain bioresources have high potential for use as adsorbents [8]. Among those, moso bamboo is recognized as one of the most popular bioresourses, and its adsorption characteristics have been the subject of many studies [8,9].

Adsorption in general, is the process of collecting soluble substances that are in solution on a suitable interface. In the past, the adsorption process has not yet been used extensively in wastewater purification but demands for a better quality of treated wastewater effluent have led to an intensive examination and use of the process of adsorption on adsorbents. Adsorbent is a very expensive adsorbent for the removal of pollutant so other inexpensive adsorbents must be investigated [10–17].

The adsorbents derived from pomegranate rind could be a potential application for the removal of nitrate. Therefore, the present study was undertaken to produce adsorbents by thermal and physical activation process utilizing pomegranate rind as abundant local raw material for application in efficient nitrate removal.

2. Materials and methods

2.1. Sample collection

Pomegranate rind was collected from a local juice manufacturing industry, Rourkela. The rind was cut into small pieces, dried in sunlight for five days and further dried in a hot air oven at 60°C for 24 h. The completely dried material was chipped and then powdered well. The powdered pomegranate rind was used for carbon preparation by thermal activation.

2.2. Pretreatment of sample and production of adsorbents

The pomegranate samples were dried at 105°C for 24 h in the oven to remove the moisture content until constant weight. Two types of activations such as boiling and thermal were conducted to produce treated pomegranate rind and thermally treated carbon, respectively. For thermal activation, the processed samples were activated in the furnace at 200, 300, and 400°C for a period of 30 min followed by crushing and sieving to size fraction of less than 150 m. For boiling activation, the raw material was boiled in distilled water at 150°C for 2 h in an oven and the treated sample was dried at 105°C until constant weight. The dried samples of untreated pomegranate rind (UPR) and adsorbents by thermal treatment were crushed and sieved to the same size fractions of the thermal treated carbons. The adsorbents were identified as the UPR for inactivated (control), Boiled pomegranate rind (BPR) for boiling treatment, and TTC 200, TTC 300, and TTC 400 for the thermal treatment at 200, 300 and 400°C, respectively. All adsorbents were stored at 4°C until use for adsorption tests.

2.3. Batch adsorption studies

The adsorbents prepared from pomegranate rind were studied for removal efficiency of nitrate from aqueous solution under different experimental conditions. 0.5 g of each adsorbents produced was added into 100 mL conical flasks containing 50 mL aqueous solution of nitrate at pH 6 with concentration of 50 mg/L. The samples were agitated in a rotary shaker at temperature of 30°C for 24 h at 150 rpm. At 1, 2, 3, 4, 5, 6, 12, 18, and 24 h interval of time, samples were withdrawn to determine the residual concentration of nitrate and its equilibrium time. The adsorbents and treated solutions were then separated by filtration. Nitrate was measured with an ion meter using the nitrate electrode (Orion 720 A + ion analyzer). The ion meter was calibrated using standard nitrate solutions before every analysis. Calibration is performed in a series of standards that prepared freshly from standard solution (0.1 M NO_3^-: Orion 720 A + ion analyzer). Ionic strength adjuster solution (Nitrate ISA: Orion720 A + ion analyzer) containing $(NH_4)_2SO_4$ was added to all solutions to ensure that the samples and standards have similar ionic strength, proper pH, and to reduce the effect of interfering ions.

For comparison, nitrate was analyzed by diatization method outlined in the Standard Methods of Examination of Water and wastewater [18] using Shimazdu UV 160 spectrophotometer. Calibration is performed in a series of standard solution prepared from stock KNO_3 solutions. Also, analyses were performed in duplicate.

2.4. Kinetic studies

In kinetic studies, 50 mL $NaNO_3$ solution (50 mg/L) and 0.5 g adsorbent were used. Batch experiment was repeated at different periods for all adsorbents. NO_3^- concentrations in supernatant have been constant after a time period. This period was accepted as equilibrium time for relevant adsorbent.

2.5. *pH studies*

In order to investigate the effects of pH on nitrate adsorption, the pH of the $NaNO_3$ solutions (50 mg/L) were adjusted to different values (2, 3, 5, 6, 7, and 10) by dilute HCl or NaOH solutions. The pH was measured by using a pH—meter. The pH adjusted solution and 0.5 g adsorbent were used in batch experiments conducted at the determined equilibrium time. The pH value providing the maximum nitrate removal was determined.

2.6. *Adsorbent dosage studies*

Adsorption of 50 mg/L of $NaNO_3$ solution by different adsorbent doses (0.25–1.0 g/50 mL) for each adsorbent was carried out at the optimum pH.

2.7. *Effect of interfering ions*

The effects of presence of sulfate, phosphate, and ammonium ion on nitrate adsorption were studied. Tests were conducted in the presence of 50 mg/L of nitrate and 10 mg/l each of sulfate, phosphate, and ammonium ions. The K_2SO_4, KH_2PO_4, and NH_4Cl salts were used to obtain the respective anions. By using various amounts of adsorbents, tap water sample (50 mL) was also used in nitrate adsorption studies. The adsorbent amounts used were 0.05, 0.1, 0.2, 0.3, 0.4, and 0.5 g. The amount of 0.2–g/50 mL tap water was found to be optimum for adsorption of nitrate.

3. Results and discussion

3.1. *Characteristics of the adsorbents*

Physical characteristics namely porosity (%), bulk density, moisture content (%), ash content (%), yield (%), and surface area of the adsorbents used in this study were determined and are depicted in Table 1 to identify the applicability of these adsorbents to remove nitrate for aqueous solution by adsorption. It was observed from the table that the porosity and surface area of TTC 400 was higher than the other adsorbents. TTC 400 was found to exhibit maximum adsorption capacity.

3.2. *Characteristics of adsorbing materials*

A scanning electron microscope (SEM) was used to examine the surface of the four adsorbents and the SEM photographs (Fig. 1) shows progressive changes in the surface of the particles. It is evident from the SEM micrograph that the materials were of irregular structure and rough surface.

3.3. *Effect of contact time*

The adsorption capacity of nitrate was observed to increase in order of TTC 400 TTC 300 TTC 200 UPC BPR as shown in Fig. 2. The concentration of nitrate by adsorbents at temperature 400°C (TTC 400) was decreased substantially from initial concentration of 50–16.5 mg/L within 1 h of treatment. The percentage removal during the first hour was around 69% at the adsorption capacity of about 3.5 mg/g. For TTC 300, the initial concentration of 50 mg/L was decreased to 36 mg/L during the first hour, while the adsorption capacity and percentage removal were 1.2 mg/g and 25%, respectively. The final concentration of nitrate adsorbed by both samples reached equilibrium point within 6 h of operation. The adsorbents UPR, BPR, and TTC 200 have reduced the nitrate concentrations of 25, 14, and 27%, respectively, from an initial concentration of 50 mg/L during the first hour of operation. Thermal treated carbons showed better adsorption capacity compared to the UPR and BPR. Only two adsorbents, TTC 300 and TTC 400 were selected for further optimization of pH and concentration of nitrate solution with the equilibrium time of 6 h.

Table 1
The physical properties of the different types of the adsorbents

Adsorbent type	Porosity (%)	Bulk density (kg/m^3)	Moisture content (% wt.)	Ash content (% wt.)	Yield (%)	Surface area (m^2/g)
UPR	41	368.44	37.15	3.35	–	385
BPR	49	402.20	24.6	6.56	–	420
TTC 200	60	584.6	6.79	9.98	90	620
TTC 300	66	275.75	9.7	32.14	56	711
TTC 400	80	275.1	1.59	58.8	46	852

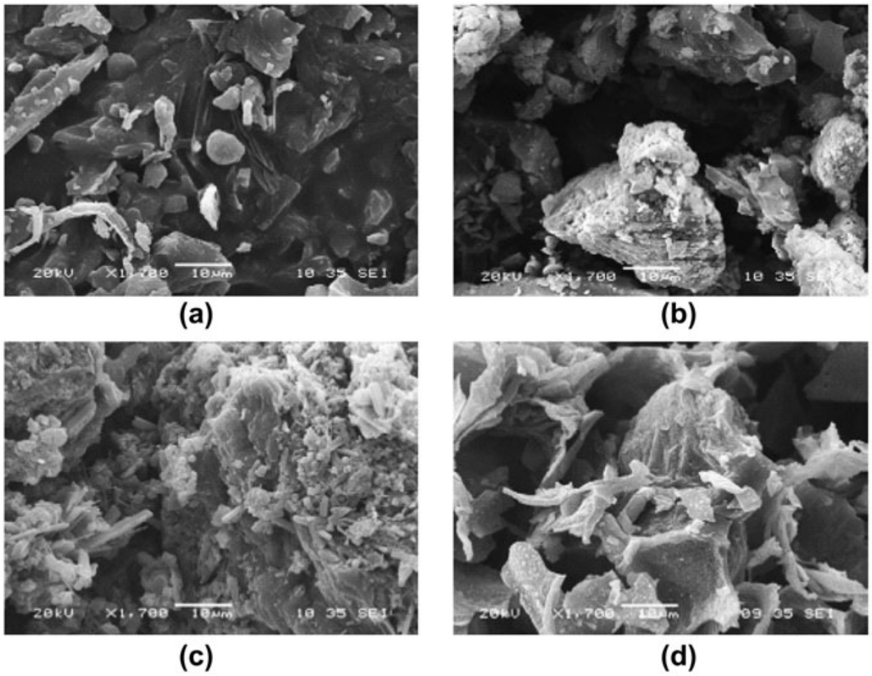

Fig. 1. Scanning electron micrographs of the four adsorbents, (a) BPC (b) TTC 200, (c) TTC 300, (d) TTC 400.

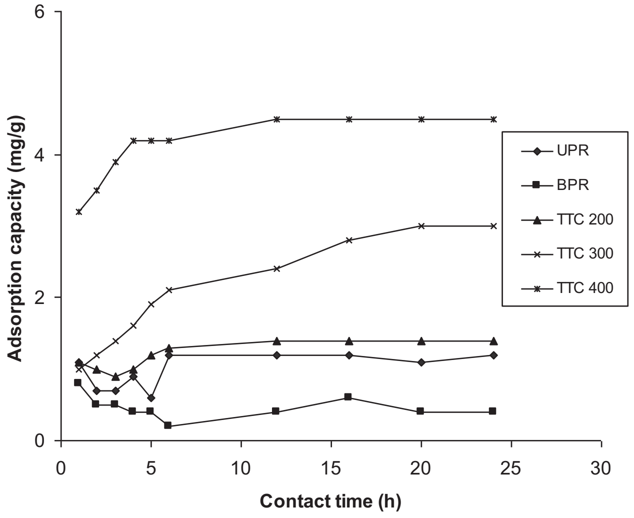

Fig. 2. Variation of contact time on the adsorption capacity of adsorbents produced from pomegranate rind in aqueous solution of nitrate. Adsorbent dosage: 1% w/v; initial concentration 50 mg/L; temperature 30˚C pH 3; agitation 150 rpm.

3.4. Effect of initial pH

The variation of pH on adsorption capacity of nitrate by adsorbents is shown in Fig. 3. The adsorption capacity of TTC 300 and TTC 400 was decreased from 2.8 to 1.7 mg/g and 4.6 to 4.2 mg/g, respectively, when the initial pH of the aqueous solution was increased from pH 2.5 to 5.5. The nitrate content remained constant in the pH range of 2–3. However, adsorption capacity for both carbons was increased to 2.2 mg/g for TTC 300 and 4.1 mg/g for TTC 400 when the pH was further increased from 5 to 8. Though there is increase in OH^- concentration at increased pH, yet the adsorption capacity of NO_3^- increases. This may probably due to the preferential adsorption of Nitrate. Among OH^- and NO_3^-, affinity of TTC 400 for NO_3^- is greater than OH^-.

3.5. Effect of initial nitrate concentration

The effect of initial nitrate concentration (25–200 mg/L) on adsorption capacity by the adsorbents TTC 300 and TTC 400 is shown in Fig. 4. Adsorbents TTC 400 had a higher adsorption capacity which varied between 2.6 and 10.5 mg/g at initial nitrate concentrations of 25–200 mg/L. TTC 300 had an adsorption capacity between 1.5 and 6.5 mg/g for the same conditions. The increase in adsorption capacity of adsorbent may be due to the higher probability of collision between nitrate and adsorbent.

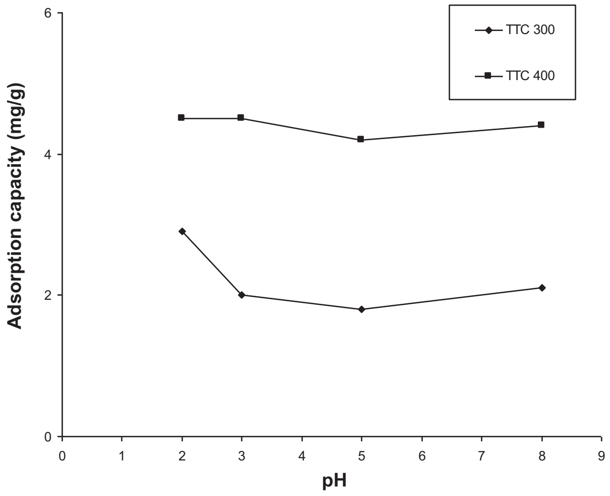

Fig. 3. Removal of nitrate by activated carbons (TTC 300 and TTC 400) with the variation of pH. Adsorbent dosage: 1% w/v; initial concentration 50 mg/L; temperature 30°C; pH 3; agitation 150 rpm; contact time 6 h.

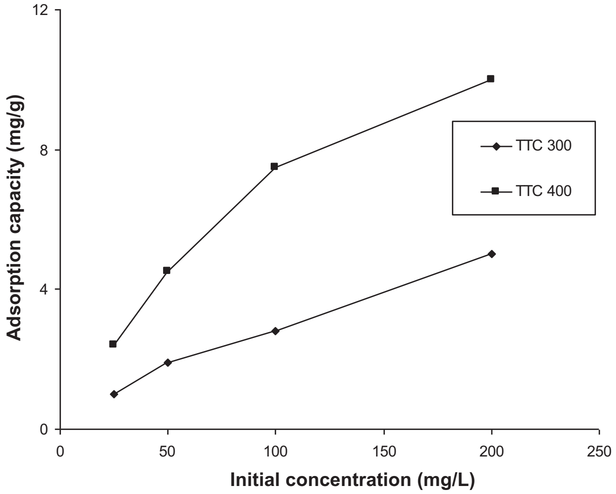

Fig. 4. Effect of initial nitrate concentration on adsorption by activated carbons (TTC 300 and TTC 400). Adsorbent dosage: 1% w/v; initial concentration 50 mg/L; temperature 30°C; pH 3; agitation 150 rpm; contact time 6 h.

Increasing the adsorbate concentration generally caused a decrease in adsorption capacity. Lower initial concentration also favored higher adsorption yield, which was the limited to 200 mg/L in this study. Further higher concentration (>200 mg/L) may reduce the adsorption capacity with the adsorbents [19].

3.6. Adsorption isotherms

The Freundlich isotherm has been adopted to characterize the adsorption capacity of organic pollutants using different adsorbents by fitting the adsorption data. The Freundlich isotherm has the general form such as:

$$q_e = K_F C_e^{1/n} \tag{1}$$

This equation can be modified as:

$$q_e = (C_0 - C_e)V/M = K_F C_e^{1/n} \tag{2}$$

where q_e is the adsorption capacity (mg/g); C_0 and C_e are the initial and equilibrium concentration (mg/L), respectively; V is the volume of synthetic nitrate solution; M is the adsorbent dose (g); and K_F and $1/n$ are the adsorption capacity and intensity of adsorption, respectively. The values of K_F and $1/n$ can be respectively determined from the intercept and slope of the logarithmic plot in Eq. (2).

$$\ln q_e = \ln K_F + 1/n \ln C_e \tag{3}$$

In order to facilitate the estimation of the adsorption capacities at various conditions, the Langmuir adsorption isotherm, a typical model for monolayer adsorption was applied. The linearized Langmuir model can be written as

$$C_e/q_e = (1/K_L q_m) + (1/q_m)C_e \tag{4}$$

where q_e is the amount of nitrate adsorbed at equilibrium (mg/g); C_e is the equilibrium concentration of nitrate (mg/L); K_L (L/mg) and q_m (mg/g) are the Langmuir constant and maximum adsorption capacity. The constants q_m and K_L can be determined from the intercept and slope of the linear plot of the experimental data of C_e/q_e vs. C_e.

Judging the correlation coefficients, R^2, compares the applicability of the isotherm equation. These adsorption isotherms were made based on the equilibrium data obtained from nitrate adsorption starting at different initial adsorbate concentrations (25–200 mg/L).

In Freundlich, a plot of ln q_e vs. ln C_e from Fig. 5 enables the empirical constant K_F and $1/n$ to be determined from the intercept and slope of the linear regression is shown in Table 2. For Langmuir, a plot of C_e/q_e vs. C_e from Fig. 6, q_m and K_L can be determined from its slope and intercept as shown in Table 2.

The correlation coefficient showed that the experimental data were better fitted in the Freundlich isotherm ($R^2 = 0.9988$ for TTC 400 and 0.9878 for TTC 300) than the Langmuir isotherm ($R^2 = 0.9796$ for TTC 400 and 0.796 for TTC 300). The Freundlich equation was more suitable when a change in the mechanism

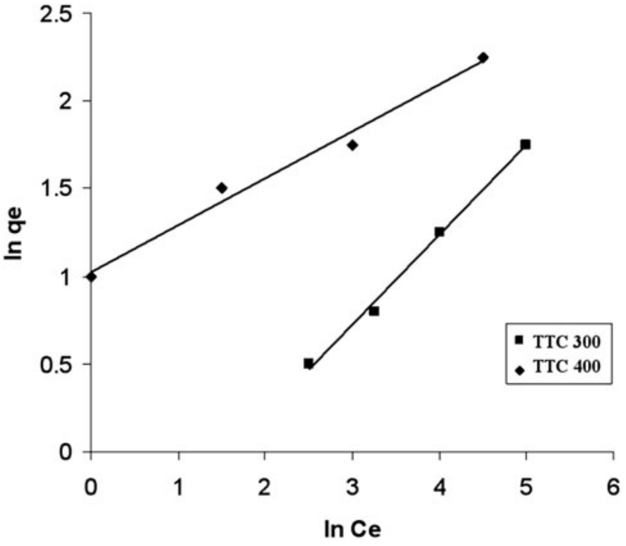

Fig. 5. Linearized Freundlich adsorption isotherm of nitrate by activated carbons (TTC 300 and TTC 400). Adsorbent dosage: 1% w/v; initial concentration 50 mg/L; temperature 30°C; pH 3; agitation 150 rpm.

Table 2
Langmuir and Freundlich isotherms parameters (TTC 300 and TTC 400) for nitrate adsorption by activated carbon

Parameters	Langmuir isotherms		Parameters	Freundlich isotherms
q_m (mg/g)	13.10	TTC	$1/n$	0.69
K_L (L/mg)	0.007	300	K_F (mg/g) (mg/L)^n	0.22
R^2	0.796		R^2	
			0.9878	
q_m (mg/g)	11.61	TTC	$1/n$	0.32
K_L (L/mg)	0.123	400	K_F (mg/g) (mg/L)^n	2.74
R^2	0.9796		R^2	

of adsorption with concentration was considered. The Langmuir equation is used for homogeneous surfaces while the Freundlich isotherm model assumes neither homogeneous site energies nor limited levels of adsorption and can result from the overlapping patterns of several Langmuir—type adsorption phenomena occurring at different sites on complex adsorbents.

3.7. Adsorption kinetics

In order to investigate the mechanism of adsorption rate for the adsorption of nitrate by adsorbents derived from pomegranate rind, the rate constants were determined by applying the equations of [20], a pseudo-first-order mechanism and [21], a pseudo-second-order mechanism, respectively. The differential equation of a pseudo-first-order kinetic is as follows:

$$(dq_t/d_t) = K_1(q_e - q_t) \tag{5}$$

Integrating Eq. (5) for the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$ gives

$$\log(q_e - q_t) = [\log q_e - (K_1 t/2.303)] \tag{6}$$

where q_e is the amount of nitrate adsorbed at equilibrium (mg/g); q_t is the amount of nitrate adsorbed at time t (mg/g) and K_1 is the equilibrium rare constant $h - 1$. In order to obtain the rate constant, the straight line plot of log $(q_e - q_t)$ vs. t for different treatment conditions was analyzed. The differential equation of a pseudo-second-order kinetic is as follows:

$$(dq_t/d_t) = K_1(q_e - q_t)^2 \tag{7}$$

Integrating Eq. (7) for the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, rearranging the final equation as:

$$t/q_t = [(1/Kq_e^2) + t/q_e] \tag{8}$$

where K is the equilibrium rate constant (g/(mg h)). The straight-line plot of t/q_t vs. t was drawn to obtain rate parameter.

The constant adsorption parameters K_1 and K for pseudo-first and second-order kinetics were determined from the plots of log $(q_e - q_t)$ against time t, and the plot of t/q_t against time t is presented in Figs. 7 and 8. The constant parameters and regression coefficients R^2 are shown in Table 3. The

Table 3
Pseudo-first- and second-order equation parameters (TTC 300 and TTC 400) for adsorption of activated carbon in aqueous solution of nitrate

Parameter	Pseudo-first-order equation	Parameter	Pseudo-second-order equation
K_1 (h^{-1})	0.70	TTC 300	3.662
R^2	0.8166	K (g/(mg h))	0.9833
		R^2	
K_1 (h^{-1})	0.832	TTC 400	0.662
R^2	0.8866	K (g/(mg h))	0.9998
		R^2	

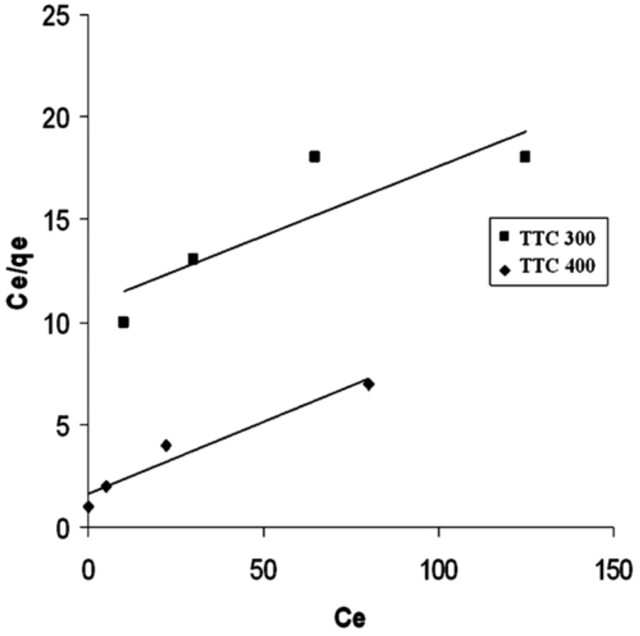

Fig. 6. Linearized Langmuir adsorption isotherm of nitrate by activated carbons (TTC 300 and TTC 400). Adsorbent dosage: 1% w/v; initial concentration 50 mg/L; temperature 30˚C; pH 3; agitation 150 rpm.

results indicated the straight lines, which showed the applicability of first- and second-order rate expression. The correlation coefficient showed that the second-order equation ($R^2 = 0.9998$) was better fitted by the experimental results compared to the first-order equation ($R^2 = 0.8866$) for adsorbents produced from TTC 400.

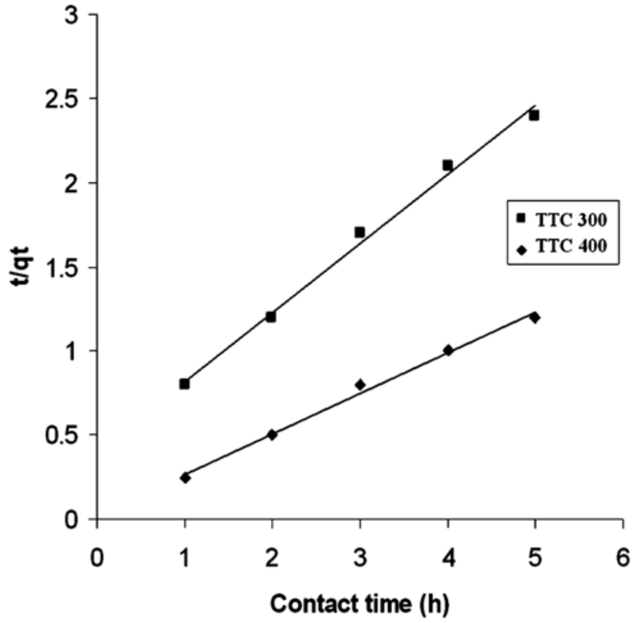

Fig. 7. Pseudo-first-order kinetic and second-order kinetic of nitrate onto activated carbons (TTC 300 and TTC 400). Adsorbent dosage: 1% w/v; initial concentration 50 mg/L; temperature 30˚C; pH 3; agitation 150 rpm.

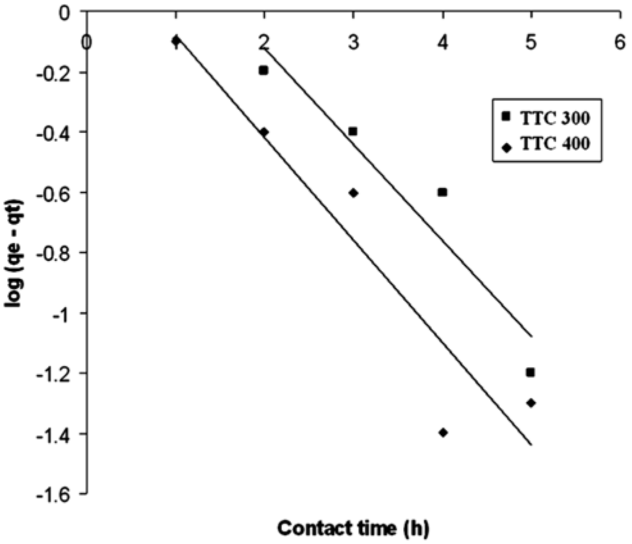

Fig. 8. Pseudo-second-order kinetic and second-order kinetic of nitrate onto activated carbons (TTC 300 and TTC 400). Adsorbent dosage: 1% w/v; initial concentration 50 mg/L; temperature 30˚C; pH 3; agitation 150 rpm.

The constant parameter K_1 in first-order kinetics indicates the nitrate adsorbed per gram of adsorbent for every hour of operation. The results indicated that 0.832 mg of nitrate was adsorbed by each gram of adsorbents TTC 400 for every hour of operations compared to the adsorbents TTC 300 (0.70 mg). While the constant parameter K for second-order kinetics equation indicates the amount of adsorbed needed in gram per unit of nitrate in each hour of operations 3.662 g of TTC 300 was needed to adsorb per mg of nitrate in 1 h of operation while only 0.662 g was required for TTC 400.

Unrinsed tamarind nutshell adsorbents and rinsed tamarind nutshell adsorbents were produced for adsorption studies and the experimental data were observed to fit Lagergren equation with the regression coefficient of 0.957 and 0.967, respectively [22]. First-order equation of Lagergren did not fit well for the whole range of contact time and was generally applicable over the initial 20–30 min of sorption process. The second-order equation is based on sorption capacity on the solid phase. It predicts the behavior over the whole range of studies supporting second-order kinetics and is in agreement with chemisorptions being the rate-controlling step [23,24].

4. Conclusion

Three adsorbents were derived from thermal activation at 200, 300, and 400˚C and one from boiling activation. The results revealed that the adsorbents at thermal activation of 300 and 400˚C showed good adsorption capacity. It was observed that the adsorption capacity decreased when the initial pH of the aqueous solution increased from pH 2.5 to 5.5. The adsorption capacity remained constant in the pH range of 2–3. However, adsorption capacity increased when the pH was further increased from 5 to 8. The correlation coefficient showed that the Freundlich isotherm ($R^2 = 0.9988$ and 0.9874 for TTC 400 and TTC 300, respectively) and pseudo-second-order kinetic ($R^2 = 0.9997$ and 0.9829 for TTC 400 and TTC 300, respectively) were better fitted than Langmuir isotherm ($R^2 = 0.9799$ and 0.7958 for TTC 400 and TTC 300, respectively) and pseudo-first-order kinetic ($R^2 = 0.8883$ and 0.8153 for TTC 400 and TTC 300, respectively). Overall, TTC 400 was found to be more efficient in removal of nitrate compared to TTC 300. The adsorbents TTC 400 was identified to be promising for the application of nitrate removal from industrial wastewater that could be encouraged for use in waste management for resource recovery and reuse.

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