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Equilibrium Studies on the adsorption of Nitrate ions onto activated carbon Prepared from Waste Cotton stalks

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*Corresponding author: Dr.V. Nandhakumar, E- mail: drvnchem2023@gmail.com ABSTRACT

Low cost adsorbents for the removal of hazardous materials are the critical requirement for the recent environmentalists. In this study, a new activated carbon was prepared from Cotton stalk using phosphoric acid in a Tubular furnace and designated as Cotton Stalk Tubular furnace Carbon (CSTC). Influence of the parameters such as dose of the adsorbent, agitation time, initial concentration of the nitrate solution, pH of the solution and temperature on adsorption of nitrate ions were studied under batch method experiments. Equilibrium data were fitted with Langmuir, Freundlich, Temkin and Dubinin-Raduskevich isotherm equations. Significance of the isotherm constant values was discussed.

KEY WORDS: Adsorption; Phosphoric acid activated Cotton stalks Carbon; Isotherms; Nitrate ions.

INTRODUCTION

Water is an integral part of all living things on the planet earth. Nitrate compounds exist in our environment due to human-made sources and also naturally. Nitrates are found in few lakes, rivers, and groundwater in Minnesota[1,2].Consuming an excessive amount of nitrate may be harmful—specifically for infants which have an effect on the incorporation of oxygen in blood and cause Methemoglobinemia (additionally referred to as blue infant syndrome)[3,4]. Bottle-fed infants beneath six months vintage are more susceptible for Methemoglobinemia. Methemoglobinemia cause bluish shadation on skin. Other signs and symptoms linked to methemoglobinemia are reduced blood pressure, elevated coronary heart rate, headaches, belly cramps and vomiting. So it is exceptionally important to remove nitrate ions from industrial waste water before discharging it into the environment [5].

Different methods are in use for the removal of various contaminants which are namely solvent extraction, membrane filtration, coagulation, chemical oxidation, chemical

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precipitation, ion exchange, reverse osmosis, membrane filtration, electro-dialysis and adsorption[6]. Most of these methods suffer from drawbacks such as high operational costs and incomplete removal or the disposal of the residual sludge formed. Adsorption has gained prominence importance than other techniques due to its simplicity in design, fast, easy to operate, relatively low operating cost and stable towards toxic substance. So that adsorption process to be a popular choice in treating wastewater [7,8].

Some of the suggested adsorbents are moss peat, fly ash, zeolite, chitosan, lignin, clay, biomass, xanthate and cactus material [9]. All the adsorbents are not economically suitable enough for wastewater treatment. Though they generally have high adsorption capacity but are expensive and difficult to be separated from the wastewater after use.

The need for cost-effective, eco-friendly and locally available alternative materials for the removal of Nitrate from aqueous solutions is essential.

Cotton (*Gossypiumhirsutum*) is one of the industrial crops which play a major role in the economics of many countries. India is the third largest country in the world which produces cotton approximately 6 x 103 tons per year. Some of the researchers tried cotton stalk as precursor for the preparation of activated carbons [10] .Therefore, cotton stalk was selected as precursor for the preparation of activated carbon in present study. Activation with H_3PO_4 generates more micro porosity and surface area which leads to an increase in the adsorption capacity [11].Hence phosphoric acid was used for preparing activated carbon in this present study.

PREPARATION OF ACTIVATD CARBON

Waste cotton stalk were collected, washed, dried and cut into small pieces. Then the materials (25 g) were mixed with 75 mL of phosphoric acid of 50% concentration and the slurry is subjected to heat treatment at 800 °C in a tubular furnace at a heating rate of 1°C min⁻¹ for 2 h in an N₂ atmosphere[12]. After that, the samples were washed thoroughly with hydrochloric acid of 5 wt. % followed with deionized water. Water wash is continued till the washing reaches the pH values 7.0. Washed product is dried at 105°C and designated as Cotton Stalk Tubular furnace Carbon (CSTC).

Estimation of Nitrate

Ion selective electrode (ISE) is an analytical technique used to determine the activity of ions in aqueous solution by measuring the electrical potential. ISE has many advantages compared to other techniques. It is relatively inexpensive and easy to operate. Wide concentration range can be measured [12].

Solubilized nitrate is determined potentiometrically using a nitrate ion-selective

electrode (ISE) in conjunction with a double-junction reference electrode and a pH meter equipped with an expanded millivolt scale (mV).

Effect of pH

Figure1 shows the effect of pH for the removal of Nitrate anions onto CSTC. The percentage of removal was found to decrease as the pH of the solution is increased. It is observed that the adsorption removal was maximum at pH 2. When pH of the solution increases, the surface of CSTC becomes negatively charged due to accumulated hydroxyl ions which prevents the adsorption of nitrate ions buy the electrostatic repulsion [13,14].



Figure 1 Effect of pH - Nitrate onto CSTC [CSTC Dose = 50 mg, Time = 70 min Ci = 80 mg/L]

Effect of adsorbent dosage

Adsorbent dosage is one of the important parameters in an adsorption process, because amount of adsorbate adsorbed vary with the dosage of an adsorbent for a given initial concentration of the adsorbate under a given set of operating conditions. Figure 2 showed the effect of adsorbent dosage on adsorption of Nitrate ions of initial concentration 80 mg/L. The percentage of nitrate ions removal increased from 32.12to 99.80%. The increase in the removal efficiency may be attributed to availability of more adsorbent surface for the nitrate ions adsorption, with an increase in the adsorbent dosage [15]. Based on these results, the remaining parts of the experiments were carried out with the adsorbent dose of 50 mg for 50 mL of adsorbate solution for Nitrate ions [16,].

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Figure 1 Effect of Dose - Nitrate onto CSTC [Nitrate ions pH = 2, Time = 70 min, CSTC Dose = 50 mg, Ci = 80 mg/L]

Effect of contact time

The contact time was evaluated as one of the most important factors affecting adsorption efficiency. The effect of contact time on the percentage removal from aqueous solution was studied by taking 40, 60, 80 and 100 mg/L of Nitrate ions as initial concentrations. The result of the above study was shown in Figure2. The rate of percentage removal was found to be rapid at initial stages and found to decrease as the contact time increases then become constant showing the attainment of equilibrium [17]. The time to attain equilibrium found to be 70 to 120 minutes for Nitrate ions for the chosen initial concentrations. At the initial stage, the ratio of surface area of the adsorbent to the amount of solute in liquid phase is high and hence the driving force makes solute to rush towards the adsorbent surface. As the time increases the above ratio begins to decrease due to adsorption and hence the rate of adsorption becomes slow [18].



Figure2 Effect of contact time for Nitrate onto CSTC [Nitrate ions pH = 2, CSTC Dose = 50 mg, Ci = 80 mg/L]

Effect of initial concentrations

The effect of initial concentration study showed that the percentage of the removal of adsorbate decreased with the increase of initial concentration of adsorbate solution as given in and shown in Figures 3 & 4. The percentage removal of Nitrate ions was found to decrease from 72.21 to 60.98, at the temperature 303 K as the initial concentration of Nitrate ions increased from 40 mg/L to 100 mg/L(Table 1). The amount of solute in the liquid phase is high at a higher initial concentration. The ratio of available adsorbent surface to the concentration of solute decreases with the increase of initial concentration. This is the reason for the decrease of percentage of removal when the concentration of the solution is increased [19]. But the amount of nitrate adsorbed was found to increase with the increase of initial concentration of the solution (Table 1). This is because increase of fraction of solute concentration transferred from liquid phase to solid surface increase with the increase of initial concentrations [20].

Table – 1 Effect of initial concentration on percentage of removal

Adsorbate	Initial Concentration C _i (mg/L)	Percentage of Removal	qe
	40	72.21	28.80
	60	68.15	40.80
Nitrate	80	64.37	51.50
ions	100	60.98	60.00

[Nitrate ions pH = 2, Dose = 50 mg/50 mL]

Effect of temperature

It is well known that temperature plays an important role in the adsorption process. The influence of temperature on adsorption of Nitrate ions were investigated at 305, 315, 325 and 335 K. Plots drawn between percentage removals versus temperature as well as amount adsorbed versus temperature were given in Figures 5& 6. It could be clearly seen that, both the percentage of removal and amount adsorbed increased with an increase of temperature. This is because; higher temperature eased the sorption by swelling of the adsorbent structure which enhanced the penetration of the big Nitrate ions[21,22].



Figure 5 Temperature vs % of Removal for Nitrate onto CSTC & Figure 6 Temp.vs Qe for Nitrate onto CSTC

[Nitrate ions pH = 2, Time = 70 min, CSTC Dose = 50 mg, C_i= 80 mg/L]

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Isotherm studies

Langmuir isotherm

The Constants obtained from Langmuir models for the removal of Nitrate ions on CSTC were given in Table2.Concerned isotherm plots were shown in Figure 8. The regression coefficient (\mathbb{R}^2) values were ranged from 0.9705 to 0.9885 for the four studied temperatures [23]. These results showed the best fitting of the equilibrium data with Langmuir isotherm. Where, Q_0 is a constant related to adsorption capacity (mg/g) and b is Langmuir constant related to energy of adsorption [24].



Figure 8 Langmuir - I Isotherm for Nitrate onto CSTC Table – 2Langmuir isotherm results

Adsorbate	Temperature (K)	Q ₀ (mg/g)	b (L/mg)	R ²
	305	121.951	0.027	0.9873
	315	136.986	0.024	0.9705
Nitrateions	325	138.889	0.025	0.9761
1 (iti attion)	335	140.845	0.026	0.9885

[Nitrate ions,	pН	= 2;	Dose =	50 mg	/ 50	mL]
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Table 3 Dimensionless separation factor (R_L)

	Initial Concentration	
Temperature (K)	Ci	$\mathbf{R}_{\mathbf{L}}$
	40	0.483383
205	60	0.384153
505	80	0.318725
	100	0.295163

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	40	0.511461
215	60	0.411053
515	80	0.343599
	100	0.287693
325	40	0.502419
	60	0.402324
	80	0.335487
	100	0.173913
	40	0.487919
335	60	0.388458
555	80	0.322681
	100	0.275953

The mono layer adsorption capacity Q_0 values (mg/g) for adsorption of Nitrate ions was ranged from 121.951 to 140.845. The adsorption capacity increased with the increase of temperature [25]. The Langmuir constant b (L/mg) values which describe the adsorption energy are very small and rules out the possibility of strong interaction between the solute and adsorption site. The dimensionless separation factor R_L values calculated for various initial concentrations at different temperatures were lie between 0 and 1 which indicate the favorable adsorption. These R_L values are presented in Table 3. In general Langmuir constant values infer a better performance of CSTC [26].

Freundlich isotherm

The values for the parameters of Freundlich model determined for the removal of Nitrate ions were given in Table 4.The Freundlich isotherm plots were shown in Figure 9.

Adsorbates	Temperature	n	K _f	R ²
	(K)		(mg/g)	
Nitrateions	305	1.6018	6.3944	0.9997
	315	1.5342	6.3797	0.9990

Table – 4Freundlich isotherm results [Nitrate ions, pH = 2, Dose = 30 mg/ 50 mL]

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Figure 9 Freundlich Isotherm for Nitrate onto CSTC

The regression coefficient (\mathbb{R}^2) values were ranged from 0.9990 to 0.9997 for the studied temperatures. It indicates that the experimental data fit well into Freundlich model.Adsorption capacity K_f (mg/g) for Nitrate anions were ranged from 6.1688 to 6.7639[27]. Further it is noticed that the adsorption capacity increased with the increase of temperature as shown in Table 4.The constant 'n' values related to adsorption intensity is ranged from 1.5442 to 1.6018 for all the studied systems, which indicates the favorable physical adsorption [28]. 'n' value increases with an increase of temperature .

Temkin isotherm

The Equilibrium binding constant K_T and the constant B_T related to heat of sorption can be determined from a plot of q_e versus lnC_e . Regression coefficient R^2 values and the Temkin isotherm parameters are summarized in table 5.Concerned isotherm plots were shown in Figure 10.The regression coefficient (R^2) values were ranged from 0.9963 to 0.9979 for the four studied temperatures viz. 305, 315, 325 and 335 K for Nitrate anions[29,30].

Adsorbates	Temperature (K)	B _T (J/mg)	K _T (L/g)	R ²
	305	118.5438	0.3733	0.9963
	315	118.7984	0.3709	0.9973
Nitratoions	325	120.2461	0.3769	0.9975
	335	121.9702	0.3873	0.9979

Table – 5 Temkin isotherm results [Nitrate, pH = 2; Dose = 30 mg/ 50 mL]



The K_T values (L/g) were ranged from 0.3709 to 0.3873. The constant B_T values are ranged from 118.5438 J/mg to 121.9702 J/mg for the four studied temperatures. The lower value of K_T and B_T indicates physisorption rather than chemisorption[31].

Dubinin-Radushkevich isotherm

The constants obtained from Dubinin-Radushkevich isotherm were presented in Table 6 and the Temkin plots are shown in Figure 11.The regression coefficient (R^2) values were ranged from 0.9329 to 0.9384 for the four studied temperatures viz. 305, 315, 325 and 335 K for Nitrate anions [32].

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Table – 6 Dubinin-Radushkevich isotherm results

Adaanhataa	Temperature	q _D	Ε	D 2
Ausorbates	(K)	(mg/g)	(kJ/mol)	K
	305	82.52	0.0005	0.9329
Nitrate ions	315	83.33	0.0005	0.9354
	325	84.31	0.0005	0.9363
	335	85.31	0.0005	0.9384

[Nitrate, pH = 2; Dose = 30 mg/ 50 mL]

The theoretical saturation capacity ' q_D ' values (mg/g) were ranged from 82.52 to 85.31. It is noticed that adsorption capacity increased with an increase in temperature [33]. The values of mean adsorption energy(E) ranged from 0.0005 kJ/mol to 0.0005 kJ/mol. These values of E are lesser than 8 kJ/mol indicating that the adsorption were physisorption [34,35].



Figure11D-R Isotherm for Nitrate onto CSTC

Conclusion

Activated carbon prepared from Cotton Stalk (CSTC) found to have good adsorption capacity for nitrate ions. Equilibrium for the adsorption was achieved in about 70 minutes for the dosage of 50 mg/50 mL of solution at room temperature (303 K) for the initial concentration of dye solutions ranging from 40 to 100 mg/L. Freundlich and Temkin isotherm represents the equilibrium adsorption data well than other Langmuir isotherms studied. The fitness of Langmuir's isotherm model gave monolayer coverage capacity of the sorbate on the surface of the adsorbent. The R_L values of Langmuir isotherms were in between 0 to 1 indicating the favourable adsorption. The adsorption energy b values were ranged from 0.024 to 0.027. It indicates the adsorption physisorption in nature.

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