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### Adsorption Of Fluoride Onto Activated Newspaper Carbon: An Equilibrium, Kinetic And Thermodynamic Study

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#### ABSTRACT

Batch sorption studies have been carried out to determine the effect of initial concentration of the sorbate, agitation time, adsorbent dose, pH, co-ions and temperature on the adsorption of fluoride on activated carbon prepared from newspaper. The adsorption process was found to be pH, temperature and concentration dependent. The experimental data fitted well to the Freundlich and Langmuir isotherms. Thermodynamic parameters such as  $\Delta H^0$ ,  $\Delta S^0$  and  $\Delta G^0$  were calculated indicating that the adsorption was spontaneous and endothermic. A mechanism in which intra particle diffusion is the rate limiting step has been proposed for the adsorption of  $F^-$  onto the adsorbent. XRD patterns of the adsorbent, before and after treatment, were recorded to get a better insight into the mechanism of adsorption process.

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#### KEYWORDS

Adsorption;  
 Defluoridation;  
 Isotherms;  
 Activated carbon;  
 Fluoride.

#### INTRODUCTION

Fluoride in drinking water is known for both beneficial and detrimental effects on health. The presence of fluoride in drinking water, within permissible limits of 1.0-1.5mg/l, is beneficial for the production

and maintenance of healthy bones and teeth, while excessive intake of fluoride causes dental or skeletal fluorosis which is a chronic disease manifested by mottling of teeth in mild cases, softening of bones and neurological damage in severe cases<sup>[4]</sup>. The fact that the problems associated with the excess fluo-

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ride in drinking water, is highly endemic and widespread, prompted many researchers to explore quite a good number of materials for defluoridation of water.

Current defluoridation methods involve precipitation and/or adsorption. Precipitation processes involve addition of chemicals, like aluminum and calcium salts<sup>[8,16]</sup>, and formation of fluoride precipitates. In adsorption method, different types of adsorbents are being used for defluoridation. Recent attention of scientists has been devoted to the study of low cost, but effective materials. A large number of materials have been tested, such as activated alumina<sup>[11,19]</sup>, brick powder<sup>[21]</sup>, magnesia<sup>[1]</sup>, plaster of Paris<sup>[6]</sup>, activated carbon, kaolinite and bentonite<sup>[17]</sup>, hydroxyapatite, fluor spar, quartz and calcite<sup>[4]</sup>, etc., and the search is still going on.

In this paper, an attempt has been made to study the equilibrium, kinetics and thermodynamics of removal of fluoride from water by a low cost adsorbent, activated newspaper carbon (NPC). The adsorption properties of activated carbons prepared from waste newspaper by chemical and physical activation were only be recently explored<sup>[15]</sup>.

### EXPERIMENTAL

#### Materials

All the reagents used were of commercially available high purity analar grade (SRL, India/sd-fine, India). Stock solutions of fluoride were prepared by dissolving sodium fluoride in doubly distilled water.

#### Preparation of activated carbon

The activated carbon was prepared by treating newspaper with 1% sulphuric acid. The material was washed with distilled water to remove excess of acid and activated at 400°C in a furnace. Grinding the material, followed by sieving gave the adsorbent. The physicochemical characteristics of the adsorbent were found out experimentally by conventional methods.

#### Methodology

The concentration of fluoride was measured using a fluoride ion-selective electrode (Orion USA, model 94-09). pH measurements were made with

systronic digital pH meter. The zero point charge of the adsorbent ( $\text{pH}_{\text{ZPC}}$ ) was calculated as described in the literature (Figure 1)<sup>[9]</sup>. The particle size of the adsorbent was determined using the Carl Zeiss light microscope (Axiostar plus) as reported earlier<sup>[1]</sup>. The XRD patterns of the adsorbent, before and after treatment with fluoride ions, were recorded at the regional research laboratory, thiruvananthapuram, S.India.

#### Batch adsorption experiments

Adsorption experiments were performed by agitating 1g of adsorbent with 50ml of fluoride solution of desired concentrations at  $30 \pm 1^\circ\text{C}$  in different stoppered bottles in a shaking thermostat machine (Tempo, India). The shaking speed was 120 strokes per min, throughout the study. At the end of pre-determined time intervals, the sorbate was filtered and the concentration of fluoride was determined. All experiments were carried out twice. Adsorbed fluoride concentrations were the means of the duplicate experimental results. Experimental variables considered were: (i) initial concentration of fluoride ions: 2-10mg L<sup>-1</sup>; (ii) contact time between adsorbent and the fluoride solution: 5-45min; (iii) pH: 3-11; (iv) dosage of the adsorbent: 0.1-4 g/50ml; (v) temperature: 30-50°C; and (vi) co-ions: Cl<sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, and HCO<sub>3</sub><sup>-</sup>.

#### X-Ray diffraction (XRD) studies

Adsorption reaction may lead to changes in molecular and crystalline structure of the adsorbent and

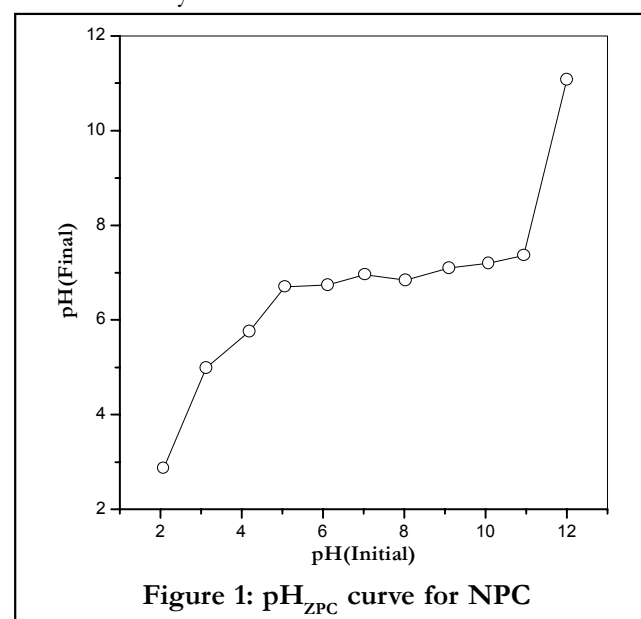


Figure 1:  $\text{pH}_{\text{ZPC}}$  curve for NPC

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hence an understanding of the molecular and crystalline structures of the adsorbent and the resulting changes thereof would provide valuable information regarding adsorption reaction. Hence, XRD patterns of the adsorbent before and after treatment with fluoride ions have been studied.

### Data analysis

The experimental data were analyzed using microcal origin(version 6) computer software. The goodness of fit was discussed using coefficient of determination,  $r^2$ , and standard deviation, sd.

## RESULTS AND DISCUSSION

### Sorbent characterization

The physicochemical characteristics of the adsorbent NPC are: particle size, 0.24-0.54 mm;  $\text{pH}_{\text{ZPC}}$ ,

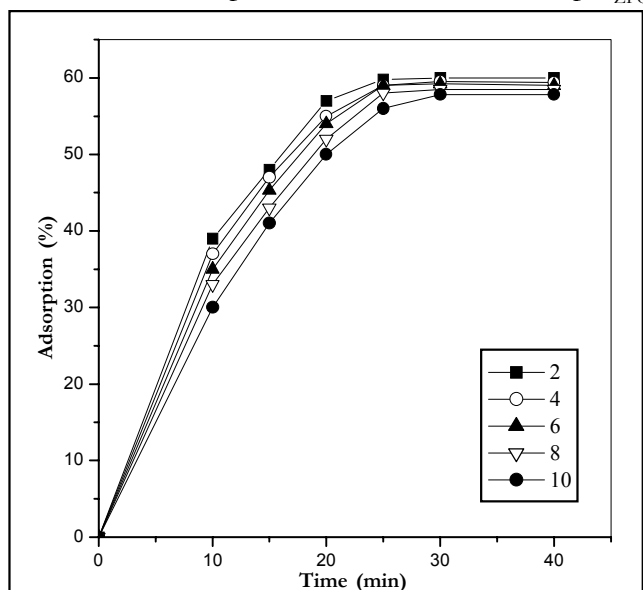


Figure 2: Effect of agitation time and initial concentration(mg/L) for the adsorption of fluoride on NPC

6.96; moisture content, 4.99% and bulk density, 0.2339g/ml.

### Effect of agitation time and initial concentration

Figure 2 shows the effect of agitation time and initial concentration on the adsorption of fluoride ions on the adsorbent. It is clear from the figure that the uptake of fluoride ions increases slowly with the lapse of time and reaches to saturation in 30min, which is fixed as the optimum contact time for other experiments. The equilibrium time was independent of initial fluoride ions concentration. The results in TABLE 1 revealed that, the amount adsorbed increases while the percentage removal decreases with the increase in initial concentration of fluoride ions. This indicates that there exists a reduction in immediate solute adsorption owing to the lack of available active sites on the adsorbent surface, compared with the relatively large number of active sites re-

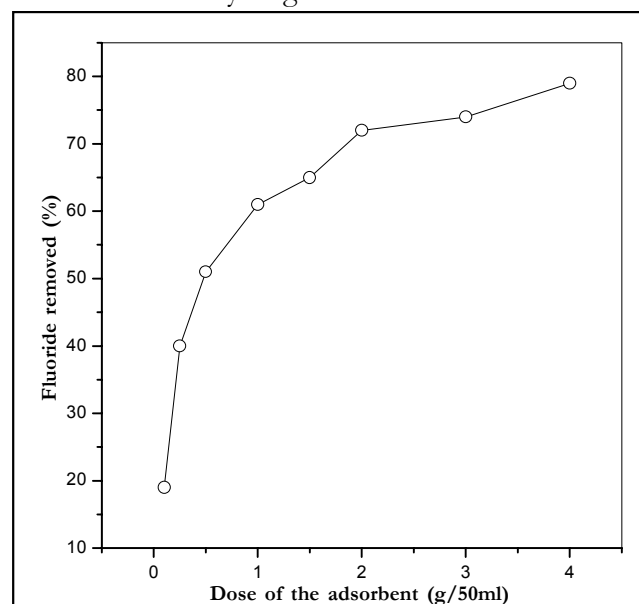


Figure 3: Effect of adsorbent dose on defluoridation of water

TABLE 1: Equilibrium parameters for the adsorption of fluoride onto NPC

[F <sup>-</sup> ] <sub>0</sub>	C <sub>e</sub> (mg L <sup>-1</sup> )			Q <sub>e</sub> (mg g <sup>-1</sup> )			Fluoride removed (%)		
	30°C	40°C	50°C	30°C	40°C	50°C	30°C	40°C	50°C
2	0.80	0.67	0.57	0.060	0.067	0.072	60.0	66.5	71.5
4	1.62	1.40	1.14	0.119	0.130	0.143	59.5	65.0	71.5
6	2.45	2.14	1.75	0.178	0.193	0.213	59.2	64.3	70.8
8	3.32	2.85	2.74	0.234	0.258	0.263	58.5	64.4	65.8
10	4.22	3.82	3.47	0.289	0.309	0.327	57.8	61.8	65.3

C<sub>e</sub>-Equilibrium concentration; Q<sub>e</sub>-Quantity adsorbed at equilibrium

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quired for the high initial concentration of fluoride<sup>[18]</sup>.

The percentage uptake is highly dependent on initial concentration of fluoride ions in the solution and it increase, at a fixed concentration of fluoride, with increase in temperature. This may be due to the fact that with increase in temperature the rate of adsorption may increase i.e. the adsorption process in endothermic in nature. Further, the fluoride ions uptake versus time curves are single, smooth and continuous leading to saturation, suggesting the possible monolayer coverage of fluoride ions on the surface of the adsorbent<sup>[13]</sup>.

### Effect of adsorbent dosage

Figure 3 shows the amount of fluoride removed as a function of adsorbent dosages at 4mg L<sup>-1</sup> of initial fluoride concentration and at 30°C. Adsorbent dosages was varied from 100mg to 4g/50ml and equilibrated for 45min. From the result it is evident that optimum adsorbent dosage of 1g/50ml is required for appreciable removal of fluoride which is fixed as the adsorbent dose for further studies.

### Adsorption isotherms

The adsorption isotherms generally used for the design of adsorption system. The Langmuir(1916) and Freundlich(1906) equations are commonly used for describing the adsorption isotherm. The linear

equation of Langmuir and Freundlich are represented as follows Eqs. (1) and (2), respectively.

$$C_e/Q_e = (C_e/Q^0) + (1/Q^0b) \quad (1)$$

$$\log Q_e = 1/n \log C_e + \log K \quad (2)$$

where  $Q_e$  and  $C_e$  has the usual meanings and  $Q^0$  and  $b$  are the Langmuir constants, indicating the adsorption capacity and energy of adsorption respectively.  $K$  and  $n$  are the empirical constants of the Freundlich isotherm measuring the adsorption capacity and intensity of adsorption respectively.

Langmuir adsorption equation was applied to quantify the adsorption capacity of the chosen adsorbent for the removal of fluoride ions from water. The adsorption isotherms were studied at 30, 40 and 50°C. The linear plots(Figure 4) of  $C_e/Q_e$  versus  $C_e$  at different temperatures indicate the applicability of the Langmuir adsorption isotherm. The value of isotherm constants and other statistical parameters were given in TABLE 2. The plots in figure 4 show that the Langmuir equation provides an accurate description of the experimental data, which is further confirmed by the high values of the coefficient of determination.

The adsorption data have been fitted to the Freundlich isotherm. The linear plots of  $\log Q_e$  versus  $\log C_e$ (Figure 5) at different temperatures indicate the applicability of Freundlich adsorption iso-

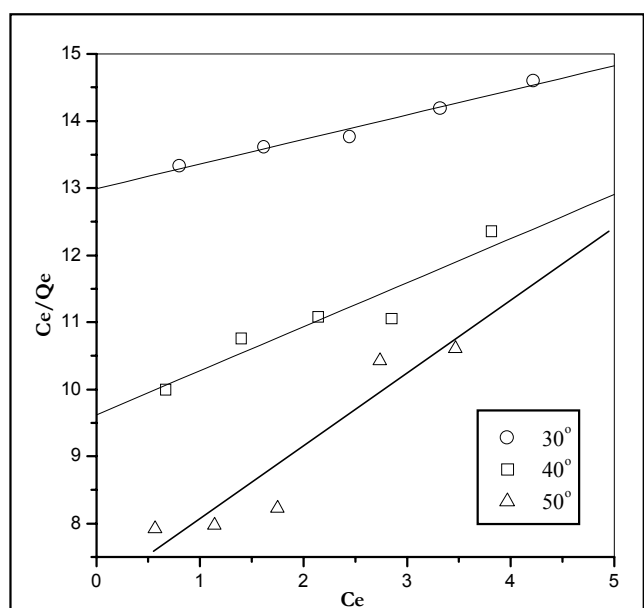


Figure 4: Langmuir plot for the adsorption of fluoride onto NPC

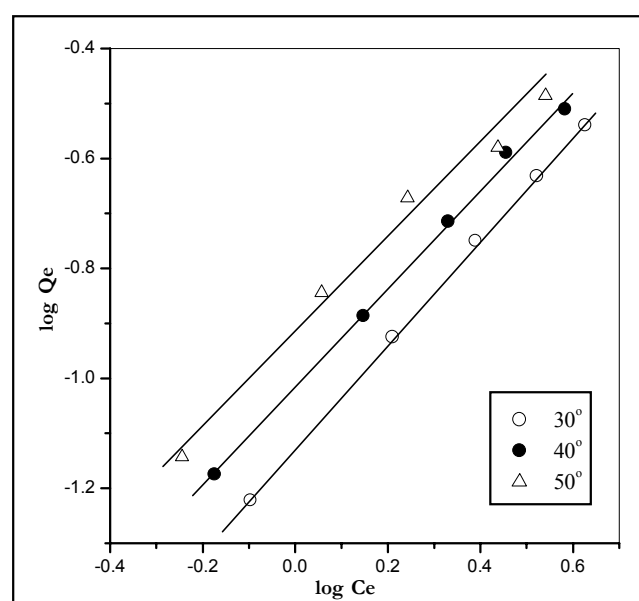


Figure 5: Freundlich plot for the adsorption of fluoride onto NPC

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therm. The results(TABLE 2) indicates that the adsorption capacity(measured by K) of the adsorbent increases with increase in temperature. Further the value of intensity of adsorption(n) is greater than unity signifies that the forces within the surface layer are attractive<sup>[10]</sup>.

The Langmuir model deals with monolayer coverage and constant adsorption energy while Freundlich equation deals with physicochemical adsorption on heterogeneous surfaces<sup>[21]</sup>. The applicability of both these isotherms to NPC, in the present study, implies that both monolayer adsorption and heterogeneous surfaces conditions exist under the experimental conditions used. The adsorption properties of NPC are thus likely to be complex, involve more than one mechanism<sup>[7]</sup>.

The essential characteristics of the Langmuir equation can be described by a dimensionless equilibrium parameter,  $R_L$ , which is defined as<sup>[13]</sup>

$$R_L = 1 / (1 + bC_0) \tag{3}$$

where b is the Langmuir constant(L mg<sup>-1</sup>) and C<sub>0</sub> is the initial fluoride ion concentration(mg L<sup>-1</sup>). The

**TABLE 2: Langmuir and Freundlich isotherm constants**

Isotherm	Statistical parameter/constants	Temperature (°C)		
		30	40	50
Freundlich	r <sup>2</sup>	0.998	0.998	0.986
	sd	0.01	0.02	0.04
	K	0.075	0.097	0.122
	n	1.05	1.12	1.22
Langmuir	R <sup>2</sup>	0.976	0.901	0.900
	sd	0.09	0.32	0.52
	Q <sup>0</sup>	2.732	1.522	0.914
	b	0.028	0.068	0.158

**TABLE 4: Rate constants for the adsorption of fluoride(10<sup>3</sup> k<sub>ad</sub> min<sup>-1</sup>) and rate constants for the forward (10<sup>3</sup> k<sub>1</sub> min<sup>-1</sup>) and reverse(10<sup>3</sup> k<sub>2</sub> min<sup>-1</sup>) processes**

[F] <sub>0</sub>	k <sub>ad</sub>		Temperature (°C)						
			30		40		50		
	30 <sup>0</sup>	40 <sup>0</sup>	50 <sup>0</sup>	k <sub>1</sub>	k <sub>2</sub>	k <sub>1</sub>	k <sub>2</sub>	k <sub>1</sub>	k <sub>2</sub>
2	6.77	7.95	13.24	4.06	2.71	5.29	2.66	9.47	3.77
4	3.39	5.83	9.51	2.02	1.37	3.79	2.04	6.80	2.71
6	3.11	3.89	9.12	1.84	1.27	2.50	1.39	6.46	2.66
8	2.42	3.32	6.36	1.41	1.01	2.14	1.18	4.18	2.18
10	1.12	1.44	3.66	0.65	0.47	0.89	0.55	2.39	1.27

value of  $R_L$  indicates the shape of the isotherms to be either unfavorable( $R_L > 1$ ), linear( $R_L = 1$ ), favorable( $0 < R_L < 1$ ) or irreversible( $R_L = 0$ ).

The  $R_L$  values computed for the present system are provided in TABLE 3. The  $R_L$  values between 0 and 1 indicate favorable adsorption for all the initial concentrations and temperatures studied. The  $R_L$  values indicated that the adsorption was more favorable for higher initial fluoride concentrations than the lower ones. Further, the sorption process becomes more favorable with increasing temperature<sup>[13]</sup>.

### Kinetic modeling

Kinetics of sorption describes the solute uptake rate which in turn governs the residence time of sorption reaction. It must be remembered that the two important physico-chemical aspects for parameter evaluation of sorption process as unit operation are the kinetics and the equilibria. Hence in the present study, the kinetics of fluoride removal has been carried out to understand the behavior of the adsorbent employed.

The sorption of fluoride from a liquid phase to solid phase may be expressed as Eqn.4.



Where  $k_1$  is the forward rate constant and  $k_2$  is

**TABLE 3: Equilibrium parameter,  $R_L$**

Fluoride concentration(mg L <sup>-1</sup> )	Temperature(°C)		
	30	40	50
2	0.947	0.880	0.759
4	0.899	0.786	0.613
6	0.856	0.710	0.513
8	0.817	0.648	0.442
10	0.781	0.595	0.388

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the backward rate constant. A represents fluoride remaining in the solution and B represents fluoride retained on the surface of the adsorbent. Since the reaction in both directions is of first order, the rate constant of adsorption,  $k_{ad}$ , was determined using the following rate expression given by Natarajan and Khalaf and described earlier<sup>[1,6]</sup>, Eqn. 5.

$$\log(C_0/C_t) = (k_{ad}/2.303) t \quad (5)$$

Where  $C_0$  and  $C_t$  are the concentration in  $\text{mg L}^{-1}$  of fluoride initially and at time  $t$  respectively.

Linear plots ( $r > 0.98$ , figure not shown) of  $\log(C_0/C_t)$  versus  $t$  suggest the applicability of the Natarajan and Khalaf equation. The rate constants were calculated from the slope and are presented in TABLE 4. The adsorption ( $k_1$ ) and desorption ( $k_2$ ) rate constants were calculated as described in the literature<sup>[1,6]</sup> and are also furnished in TABLE 4. It is evident, from the results, that the forward rate constant is much higher than the backward rate constant suggesting that the rate of adsorption is clearly dominant.

The values of  $k_{ad}$  were found to decrease with increase in the initial concentration of fluoride from 2 to  $10 \text{ mg L}^{-1}$ . An examination of the effect of fluoride ion concentration on the  $k_{ad}$  helps to describe the mechanism of removal taking place. In cases of strict surface adsorption, a variation of rate should be proportional to the first power of concentration.

However, when pore diffusion limits the adsorption process, the relationships between initial solute concentration and the rate of reaction will not be linear. Hence, it seems likely that pore diffusion limits the overall rate of fluoride adsorption<sup>[6]</sup>.

In adsorption studies, it is necessary to determine the rate-limiting step. The results obtained from the experiments, therefore, were used to study the rate-limiting step. Since during the experiment the particles were agitated vigorously, it is reasonable that the mass transfer from the bulk liquid to the external surface of the adsorbent did not limit the rate. Then, one might postulate that the rate-limiting step might be film or intra-particle diffusion. The possibility of intra-particle diffusion was tested by plotting the graph between amount of fluoride adsorbed and square root of time (Figure 6). The initial curve portions, of these curves, are attributed to boundary layer diffusion effect and the final linear portions are due to intra-particle diffusion effect<sup>[1,6,21]</sup>. In figure 6, the linear portions of the curves do not pass the origin. This indicates that the mechanism of removal of fluoride by NPC is complex and both the surface adsorption as well as intra-particle diffusion contributes to the rate-determining step<sup>[21]</sup>.

### Thermodynamic parameters

The standard free energy change ( $\Delta G^0$ ), enthalpy change ( $\Delta H^0$ ) and entropy change ( $\Delta S^0$ ) were calculated from the variation of the thermodynamic equilibrium constant,  $K_0$ ;  $K_0$  for the adsorption process

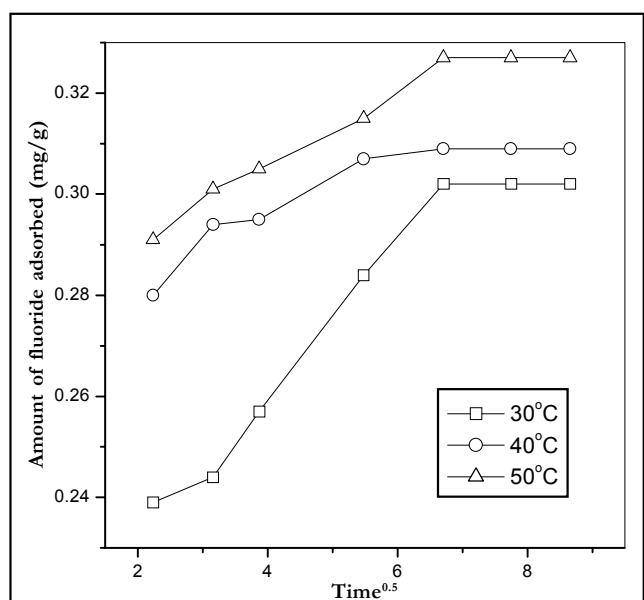


Figure 6: Plots of amount of fluoride adsorbed versus time for intra-particle diffusion of fluoride

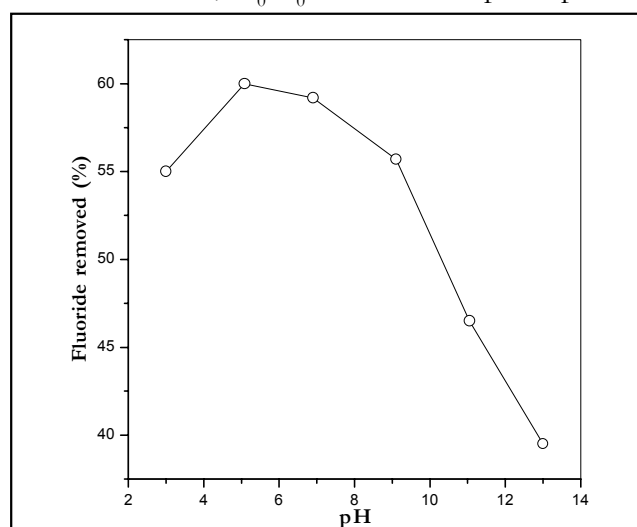


Figure 7: Effect of pH of the medium on the adsorption of fluoride onto NPC

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was determined by the reported method<sup>[18]</sup>. The thermodynamic parameters were calculated using the following equations.

$$\ln K_0 = (\Delta S^0/R) - (\Delta H^0/RT) \quad (7)$$

$$\Delta G^0 = -RT \ln K_0 \quad (8)$$

$\Delta H^0$  and  $\Delta S^0$  were determined from the slope and intercept of the plot (not shown) of  $\ln K_0$  versus  $1/T$  and were given in TABLE 5. The endothermic nature of adsorption is indicated by an increase in  $K_0$  with rise in temperature. The results in TABLE 5 indicate that  $\Delta G^0$  values are negative which mean that the reaction is spontaneous. The values of enthalpy of a sorption process may be used to distinguish between chemical and physical sorption<sup>[9]</sup>. For chemical sorption, enthalpy values range from 83 to 830 kJ mol<sup>-1</sup>, while for physical sorption they range from 8 to 25 kJ mol<sup>-1</sup>. On the basis of the above distinction, fluoride ion sorption by NPC is a physical process. Positive values of  $\Delta H^0$  suggest that the process is endothermic, so an increase of temperature encourages fluoride ion adsorption. As indicated in TABLE 5,  $\Delta S^0$  values for the adsorption process are positive. This observation suggests a high degree of disorderliness at the solid-solution interface during the adsorption of fluoride onto NPC. This may be due to the fact that the adsorbed water molecules, which are displaced by the adsorbate species, gain more translational entropy than is lost by the adsorbate molecules, thus allowing the prevalence of randomness in the system<sup>[14]</sup>.

### Effect of pH

The effect of initial pH of the fluoride solution on the amount of fluoride ions adsorbed was studied and it is depicted in figure 7. The results indicate that the adsorbent exhibits a commendable

defluoridation capacity in wide range of pH. The value of zero point charge ( $\text{pH}_{\text{ZPC}} = 6.96$ ) suggests the presence of some weakly acidic groups on the surface of the adsorbent. Hence, the defluoridation capacity of the adsorbent is appreciable in acidic range. However, at very high pH the defluoridation capacity decreases sharply. This may be due to the competition between hydroxide and fluoride ions in this pH range. Similar trend in the effect of pH of the medium on defluoridation capacity of different adsorbents were reported earlier<sup>[1,6,11]</sup>.

### Effect of co-ions

The effect of added co-ions viz.  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$  and  $\text{HCO}_3^-$  on the percentage of adsorption is depicted in figure 8. The results indicate that addition of co-ion decrease the amount of fluoride ions removed. This is due to the competition between the anions and fluoride ions for the active sites on the adsorbent<sup>[6,11]</sup>.

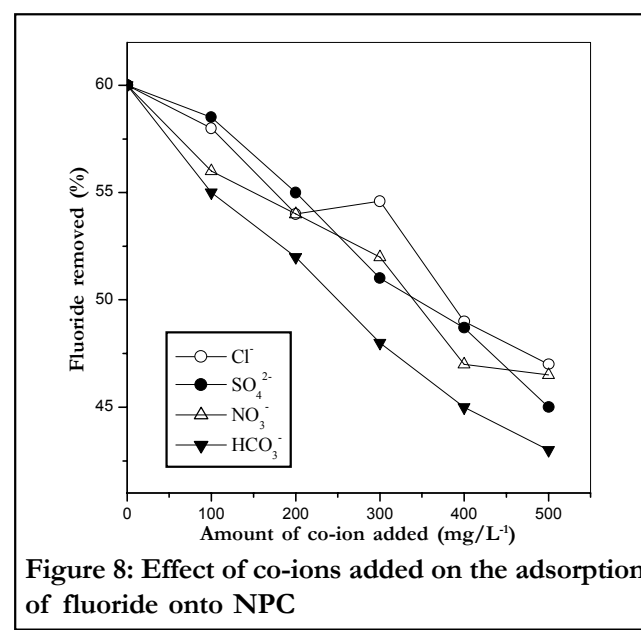


Figure 8: Effect of co-ions added on the adsorption of fluoride onto NPC

TABLE 5: Equilibrium constant ( $K_0$ ) and thermodynamic parameters for the adsorption of fluoride onto NPC

[F] <sub>0</sub>	$K_0$			$-\Delta G^0$		$\Delta H^0$	$\Delta S^0$	
	30°C	40°C	50°C	30°C	40°C			
2	1.50	1.99	2.51	1.02	1.78	2.47	20.38	70.64
4	1.47	1.86	2.50	0.97	1.61	2.47	21.13	72.78
6	1.45	1.81	2.43	0.93	1.55	2.38	20.38	70.20
8	1.41	1.80	1.92	0.87	1.54	1.75	12.34	43.80
10	1.37	1.62	1.88	0.79	1.25	1.70	12.58	44.12

$\Delta G^0$  kJ mol<sup>-1</sup>;  $\Delta H^0$  kJ mol<sup>-1</sup>;  $\Delta S^0$  J K<sup>-1</sup> mol<sup>-1</sup>

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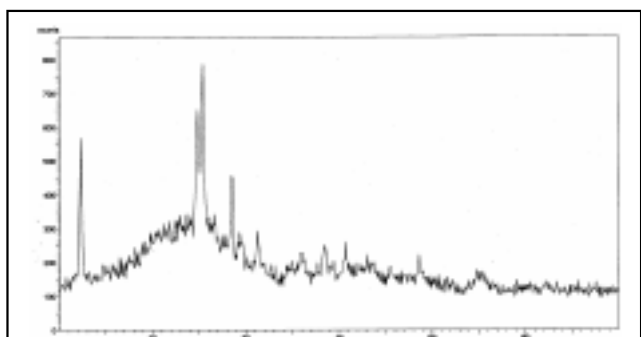


Figure 9a: XRD pattern of newspaper carbon before adsorption

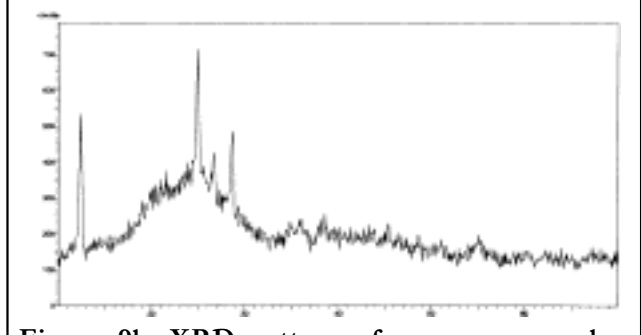


Figure 9b: XRD pattern of newspaper carbon loaded with fluoride ions

TABLE 6: Langmuir isotherm parameters for adsorption of fluoride from aqueous solution on different adsorbents

Adsorbent	Langmuir constants		Reference
	Q <sub>0</sub> (mg/g)	b (l/mg)	
Activated alumina	2.41	0.31	[19]
Fluorspar	1.79	0.091	[4]
Activated quartz	1.16	0.086	[4]
Hydroxy apatite	4.54	2.44	[4]
Calcite	0.39	0.023	[4]
Quartz	0.19	0.12	[4]
Titanium rich bauxite	3.70	0.29	[2]
Plaster of Paris	0.366	0.830	[6]
NPC	2.732	0.028	Present work

### XRD studies

The XRD patterns of the adsorbent before and after treatment with fluoride ion solution are shown in figure 9. It is evident from the XRD study that the crystal structure of the adsorbent showed no significant variation in the crystal structure after the adsorption of fluoride ions. This suggests that the

fluoride ions might diffuse into micropores and macropores and sorb mostly by physisorption without altering the structure of the adsorbent<sup>[4,20]</sup>. The above observation corroborated well with batch sorption experiments and thermodynamic results.

### Comparison with other adsorbents

Adsorption isotherms for fluoride in aqueous solution were compared for different adsorbents and the values of Langmuir parameters for the adsorption of fluoride in different adsorbents used in the literature with the adsorbent of the present study are summarized in TABLE 6. Although direct comparison of NPC with other adsorbents is difficult, owing to the different applied experimental conditions, it was found, in general, that the adsorption capacity of NPC for fluoride is comparable with that of other adsorbents and in fact greater than certain adsorbents reported earlier.

### Test with field samples

The utility of the adsorbent has been tested by treating natural water samples collected from tube well water sources located at Kumaran Thozhu (Sample 1) and Vellaiya Thevanpatti (Sample 2) of theni district, tamil-nadu, S.India. Compositions (all in mg/l except pH) of the waters are, respectively, pH: 7.6, 7.7; total alkalinity: 680, 316; total hardness: 420, 1350; total dissolved solids: 1680, 3050; NO<sub>3</sub><sup>-</sup>: 8, 15; Cl<sup>-</sup>: 300, 1100; F<sup>-</sup>: 1.9, 2.4; SO<sub>4</sub><sup>2-</sup>: 120, 175. The percentage removal of fluoride, by 1 g adsorbent per 50ml of water sample at 30°C, from samples 1 and 2 were found to be 61 and 68 respectively which is in close agreement with the results mentioned above.

## CONCLUSION

The results of the present study show that NPC has considerable potential for the removal of fluoride ions from aqueous solutions. Wide range of pH and high temperature were found as the optimum conditions for maximum fluoride adsorption by the adsorbent. The results gained from this study were extremely well described by the theoretical Freundlich and Langmuir isotherms. Values of equilibrium parameter, R<sub>1</sub>, indicated the F<sup>-</sup>/NPC system



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was favorable. The thermodynamics of the system pointed out the system was spontaneous and endothermic. The low enthalpy change for the adsorption process and XRD studies indicate that the adsorption occurs through physisorption. On the basis of the kinetic studies a mechanism has been proposed for the adsorption of  $F^-$  onto NPC in which the pore diffusion is the rate-limiting step.

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