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Data of Characterization and Adsorption of Fluoride from Aqueous Solution by Using Modified *Azadirachta Indica* Bark

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Abstract

This data was decisive on the adsorption of fluoride by microwave assisted carbonized *Azadirachta indica* bark (MACAIB) adsorbent material from aqueous solution. *Azadirachta indica* bark is a plant-based effortlessly available item which is transformed into a carbonaceous adsorbent material and utilized for the removal fluoride from aqueous solution. Characterization of the MACAIB adsorbent material demonstrated that it was porous and extremely effective in the removal of fluoride. The operating parameters such as pH, adsorbent dose, agitation speed, initial fluoride concentration, contact time and temperature were efficient on the adsorption ability of fluoride. The maximum removal efficiency of fluoride with an initial fluoride concentration 2 mg/L was found to be 83.50%. Experimental adsorption isotherm equilibrium data furnished was the best with Langmuir adsorption isotherm model, showing monolayer adsorption on a homogenous surface (most extreme monolayer adsorption capacity was 0.923 mg/g at 303 K). The adsorption kinetics experiment was followed by pseudo second-order kinetic model that indicated chemisorptions process. Intra-particle diffusion mechanism was not the sole rate-controlling factor. Thermodynamic analysis proposes that removal of fluoride from aqueous solution by MACAIB material was an exothermic and spontaneous process. Characterization of the MACAIB carbon material before and after adsorption through FTIR, SEM, EDX and XRD techniques confirmed the fluoride adsorption on the adsorbent surface. It could be accomplished that MACAIB is an effective adsorbent material for successful removal of fluoride from aqueous solution.

Keywords : Characterization, adsorption, fluoride, kinetics, isotherms, thermodynamics

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Specifications Table

Subject area	Chemistry, Environmental Sciences
More specific subject area	Adsorption
Type of data	Table, image, graph, figure
How data was acquired	SEM (JEOL Model JSM - 6390LV), EDX (JEOL JSM-7600F FEG-SEM model), FT-IR(Thermo Nicolet, Avatar 370), XRD (Bruker AXS D8 Advance difractometer model), BET analyser (Quanta Chrome Nova-1000 surface analyser) , CHNS analyser (Elemental Vario EL III model), Ion selective electrode (HANNA Model No. HI 4522), pH meter (ELICI LI 120)
Data format	Analyzed
Experimental factors	The developed adsorbent from *Azadirachta indica* bark characterized and performed its capability for removal of fluoride from aqueous solution.
Experimental features	Development of adsorbent, characterization by ultimate, proximate and instrumental analysis, performance of adsorbent for removal of fluoride by adsorption method.
Data source location	Dr. Ambedkar College, Deeksha Bhoomi, Nagpur, India
Data accessibility	Data is accessible with this article
Related research article	N. G. Telkapalliwar, V. M. Shivankar , Adsorption of Zinc onto Microwave assisted carbonized Acacia nilotica bark, Material Today Proceedings, 5 (2018) 22694–2270. [1]

Value of the Data

- Data showed that the modification of conventional absorbent can be used to remove fluoride with greater enhancement.
- Characterization data of the MACAIB is useful for the scientific community to complete studies related to fluoride adsorption.
- Data of isotherms, kinetics and thermodynamics is informative for predicting and modeling of the adsorption of fluoride from aqueous solution by MACAIB.
- Data of this project can be used to improve drinking water quality.

1. Data

The FT-IR, SEM, EDX and XRD results of removal of fluoride before and after fluoride adsorption from aqueous solution are shown in Fig. 1 to Fig. 5. The effects of operational parameters such as pH (2-4), agitation speed (20-180 strokes/min), an adsorbent dose (1-6 g/L), initial fluoride concentration (2-10 mg/L), contact time (30-300 minute) and temperature (303- 333 K) of MACAIB on fluoride adsorption are presented in Fig. 6 to Fig.11

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respectively. The linear plots of four well known adsorption isotherms viz. Langmuir, Freundlich, Temkin and Dubinin-Radushkevich models are presented in Fig. 12 to Fig.15 respectively. The rate and kinetics of fluoride adsorption onto the MACAIB material was studied via, pseudo first -order, pseudo second-order, Intra-particle diffusion and Elovich kinetic equation based models presented in Fig. 16- Fig. 19 respectively. The data of change in enthalpy (ΔH) and change in entropy (ΔS) are estimated from the intercept and slope of a direct plot between Log K versus 1/T, **Fig. 20**. The obstruction of different counter ions on fluoride removal by MACAIB material was investigated and the results acquired were plotted as appeared in **Fig. 21**. The physico-chemical characterization data of MACAIB adsorbent material is illustrated in Table 1 and Table 2. FT-IR absorption bands and possible functional groups of MACAIB before and after fluoride adsorption are presented in Table 3. The EDX analysis data of MACAIB before and after fluoride adsorption is illustrated in Table 4 and 5. The four well known adsorption isotherms and kinetic equations are presented in Table 6. The adsorption isotherms and kinetic data for removal of fluoride onto MACAIB material are presented in Table 7 and Table 8. The thermodynamic parameters calculated in the investigation are presented in **Table 9**. It was discovered that change in enthalpy (ΔH) varied from −28.452 to −17.591 kJ mol^{-1} in the initial fluoride concentration range of 2 – 10 mg/L, while the change in entropy (ΔS), varied from −0.081 to −0.066 $kJmol^{-1}$ K^{-1}.

2. Experimental Design, Materials, and Methods

2.1. Preparation of Adsorbent

Azadirachta indica bark sample was collected from the local village area and cut into small pieces. The dried Azadirachta indica bark powder was carbonized on muffle furnace for 5 Hours at 500^0C. This carbonized bark powder was again activated in domestic microwave (900MW) with an interval of one minute for 30 successive minutes. The microwave assisted carbonized bark was then impregnated with 0.5 N sodium hydroxide and afterwards with 0.5 N sulphuric acid for 24 hours separately. The resultant carbon was washed with double distilled water until the point when a steady pH of the slurry was reached. The subsequent material was dried at 110^OC in vacuum oven for 24 hours; grinded well and kept in air-tight plastic bottles for further use. The carbon material obtained from the bark of *Azadirachta indica* was later referred as microwave assisted carbonized *Azadirachta indica* bark **(MACAIB).**

2.2. Characterization of Adsorbent

The physico-chemical characterization such as proximate, ultimate and instrumental analysis of the MACAIB adsorbent were performed by BET surface analyser, CHNS elemental analyser, FT-IT, SEM, EDX and XRD [2-10].

2.3. Fluoride adsorption experiments

Batch adsorption experiments were carried out to find the adsorption capacity of MACAIB at different fluoride concentrations ranging from 2 mg/L to 10 mg/L. Samples of 50 ml each of fluoride solution of a particular concentration were shaken at 120 strokes/min for predetermined pH, adsorbent dose, contact time and temperature. The initial and final fluoride concentrations of the solutions were measured by using an ion selective meter

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(HANNA Model No. HI 4522) and Fluoride ion selective electrode (HANNA Model No. HI 4110). The fluoride adsorption capacities of the MACAIB were calculated using the equation (1):

$$q_e = \frac{(Co - Ce)\, V}{m} \quad (1)$$

Where m is the mass of adsorbent (g), V is the volume of the solution (L), C_0 is the initial fluoride concentration (mg/L), C_e is the equilibrium fluoride concentration (mg/L) and q_e is the fluoride quantity adsorbed at equilibrium (mg/g). The percent removal of fluoride from the aqueous solution was evaluated by the equation (2):

$$\%\ \text{Fluoride Removal} = \frac{(Co - Ce)}{Co}\ X\ 100 \quad (2)$$

The effects of pH, adsorbent dose, agitation speed, initial fluoride concentration, contact time and effect of temperature are studied for removal of fluoride from aqueous solutions by using MACAIB carbon material. Adsorption isotherm, adsorption kinetic and thermodynamic study was performed by varying respective parameters for adsorption of fluoride on MACAIB material by Batch adsorption experiments. Finally, desorption of fluoride and effect of different counter anions was studied for fluoride removal on MACAIB.

2.4. Adsorption isotherms

The adsorption isotherms are a standout amongst the most important methods for demonstrating the adsorption capacity of the adsorbent material and the mechanism of the adsorption system. This system expresses the precise connection between the concentration of fluoride adsorbate and its degree of adsorption onto adsorbent surface. The fluoride adsorption equilibrium records of MACAIB at four different temperature (303, 313, 323 and 333 K) have been analyzed by using four surely understood isotherm models, viz. Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (**Table 6)** [1]. It recognized that the experimental data fitted well to all these adsorption isotherm models. Correlation coefficients (R^2) values demonstrated that Langmuir adsorption isotherm gives a decent model for the adsorption of fluoride on MACAIB material which depends on monolayer adsorption on to the surface limiting a finite number of identical adsorption sites. The appropriateness of the adsorption information to the Langmuir isotherm model suggested that the binding energy on the whole surface of the MACAIB adsorbent material was uniform and that adsorbate-adsorbate interaction was small. The values of various constants of four adsorption isotherm models were determined and presented in **Table 7**.

2.5. Adsorption kinetics

The kinetic investigation of adsorption of fluoride from aqueous solutions plays a significant role because it shows crucial knowledge insight into the reaction pathways and mechanism of the fluoride adsorption process. The rate and kinetics of fluoride adsorption onto the MACAIB material was studied via, pseudo 1st -order, pseudo 2nd -order, Intra-particle diffusion and Elovich kinetic equation based models. The observed kinetic equations and kinetic parameters of adsorption kinetic models are discussed in **Table 6** [1]. The adsorption kinetics experiments followed

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pseudo second-order kinetic model indicating to chemisorptions process. It is noted that Intra-particle diffusion mechanism was not the sole rate-controlling factor.

2.6. Thermodynamic study

The Thermodynamic investigation is significantly more valuable as it gives powerful information on doing the adsorption process. Thermodynamic parameters of the adsorption system, for example, change in free energy (ΔG), change in enthalpy (ΔH) and change in entropy (ΔS) were determined at four different temperatures for initial fluoride concentrations 2 mg/L - 10 mg/L by using the equations 19, 20, 21. The Gibbs free energy (ΔG) for fluoride adsorption by MACAIB adsorbent material at all temperatures is obtained by using equation 19.

$$\Delta G = -R\,T\,\ln K \tag{19}$$

$$\Delta G = \Delta H + T\,\Delta S \tag{20}$$

$$\ln K = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \tag{21}$$

Where, K represents adsorption equilibrium constant, T is Temperature in Kelvin and R is a Universal gas constant (8.314 J $K^{-1}mol^{-1}$). A linear plot of Log K versus 1/T can be applied to find out the values of ΔS and ΔH from the slope and intercept. The values of change in enthalpy (ΔH) and change in entropy (ΔS) are estimated from the intercept and slope of a direct plot between Log K versus 1/T (**Figure 21)**. The thermodynamic parameters calculated in the present study are presented in **Table 9**. Thermodynamic analysis proposes that removal of fluoride from aqueous solution by MACAIB material was an exothermic and spontaneous process.

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Acknowledgments

The authors are thankful to the Principal, Dr. Ambedkar College, Deekshabhoomi, Nagpur for providing facilities to carry out this research work. The authors also acknowledge support from The Director, STIC, Cochin University of Science and Technology, Cochin for providing CHN analyser, SEM, FTIR and XRD facilities; SAIF, IIT, Bombay for providing EDX facility and Bangalore Institute of Technology, Bangalore for BET surface analysis.

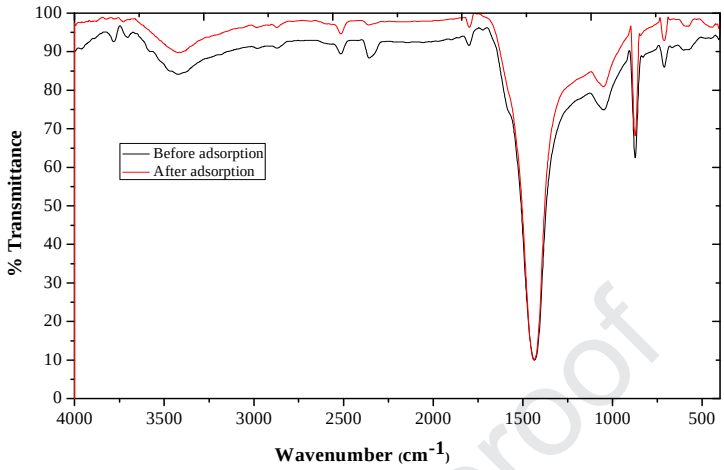
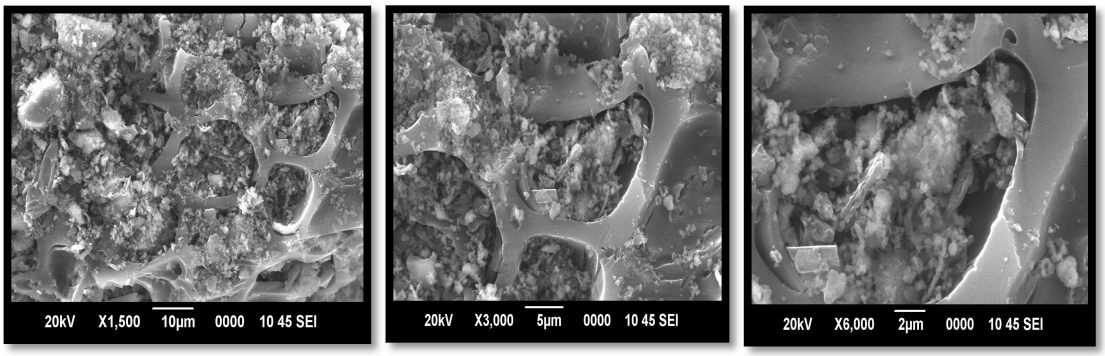
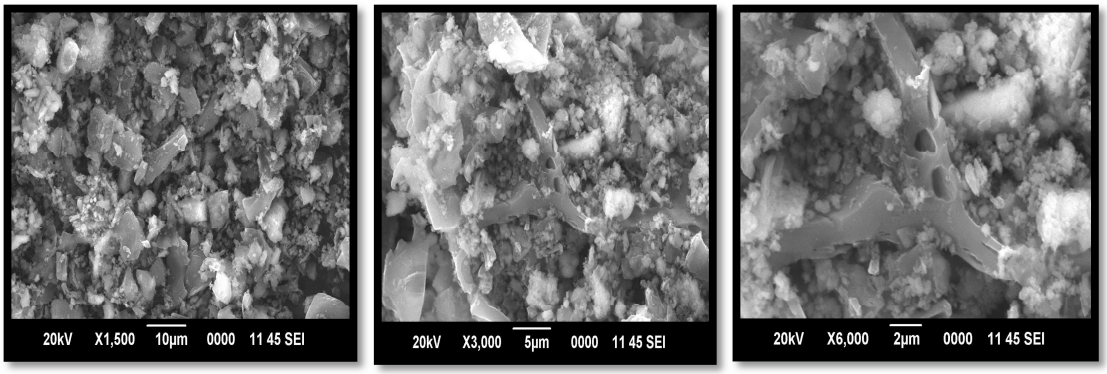


Fig. 1 : Fourier Transform Infrared (FTIR) spectra of MACAIB before and after fluoride adsorption.



(a) (b) (c)

Figure 2 (a, b, c) : SEM micrographs (10, 5 and 2 µm) of MACAIB before fluoride adsorption.



(a) (b) (c)

Fig. 3 (a, b, c) : SEM micrographs (10, 5 and 2 µm) of MACAIB after fluoride adsorption.

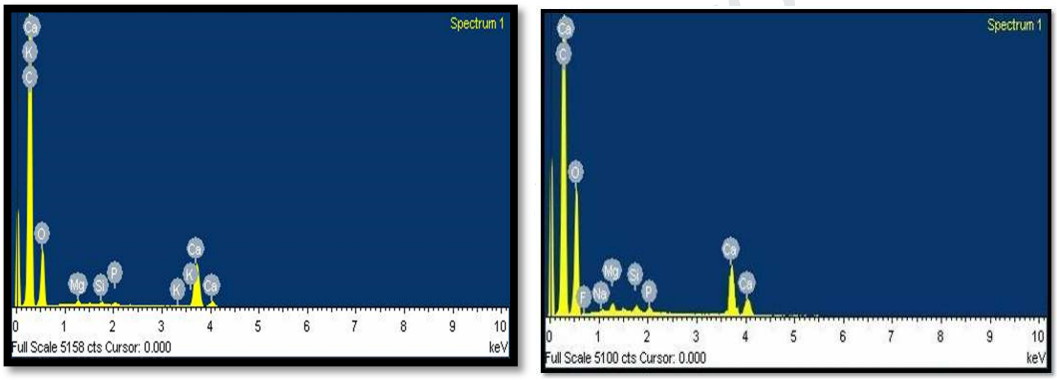


Fig. 4 : EDX monograph of MACAIB before and after fluoride adsorption.

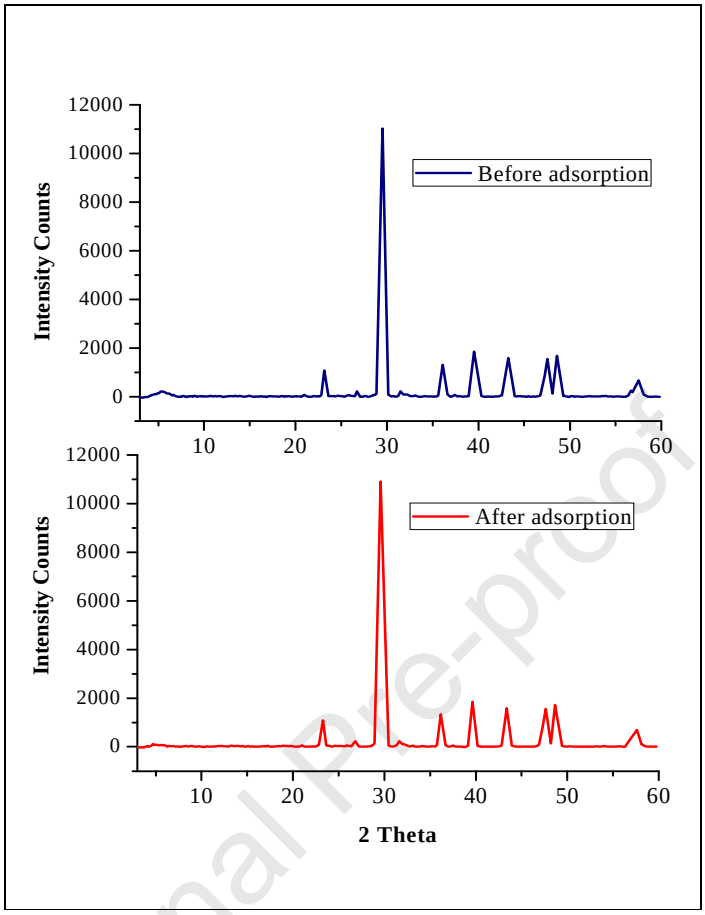


Figure 5 : Powder X-ray diffraction (XRD) of MACAIB before and after fluoride adsorption.

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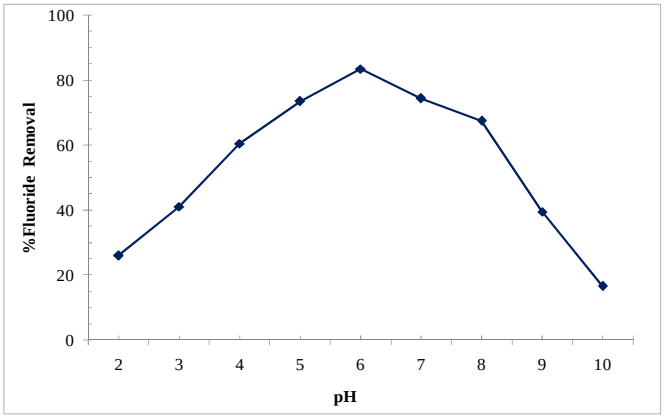

Figure 7: Effect of pH on fluoride adsorption by MACAIB

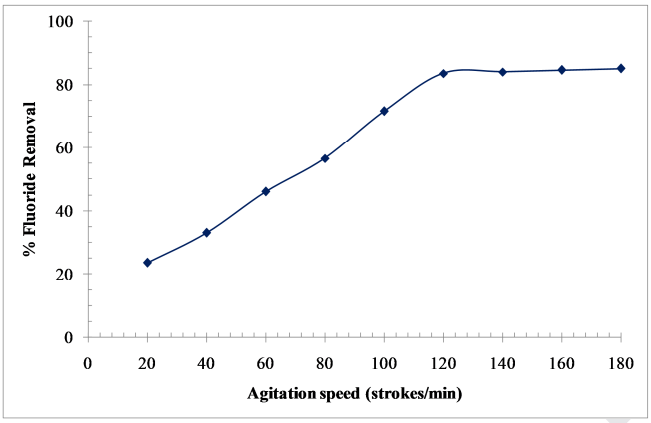

Figure 8: Effect of agitation speed on fluoride adsorption by MACAIB

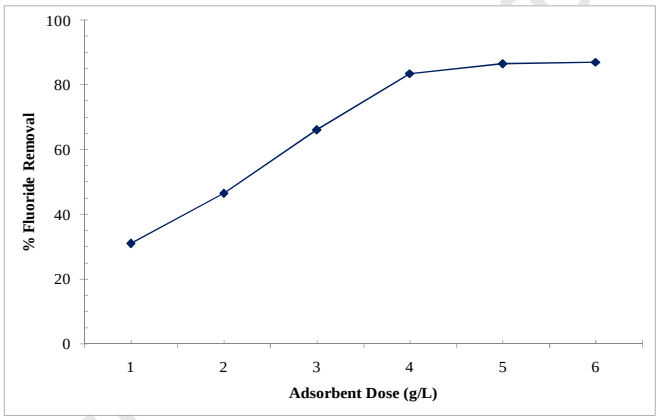

Figure 9 : Effect of adsorbent dose of MACAIB on fluoride adsorption.

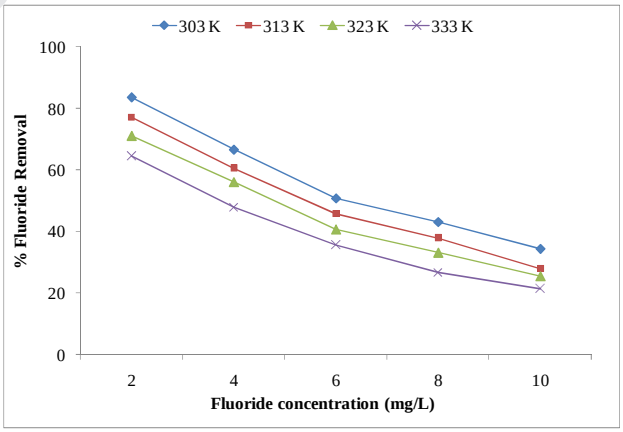

Figure 10: Effect of fluoride concentration on its adsorption by MACAIB.

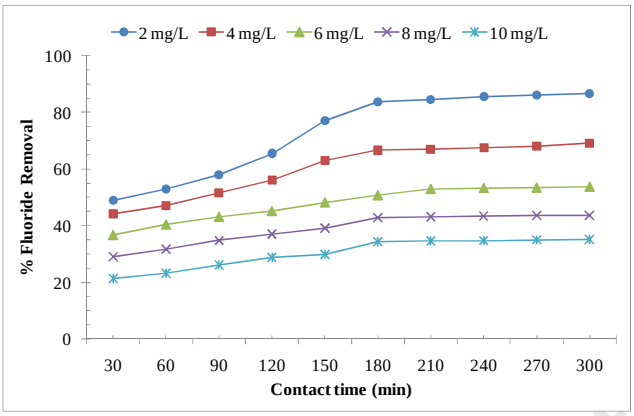

Figure 15: Effect of contact time on fluoride adsorption by MACAIB.

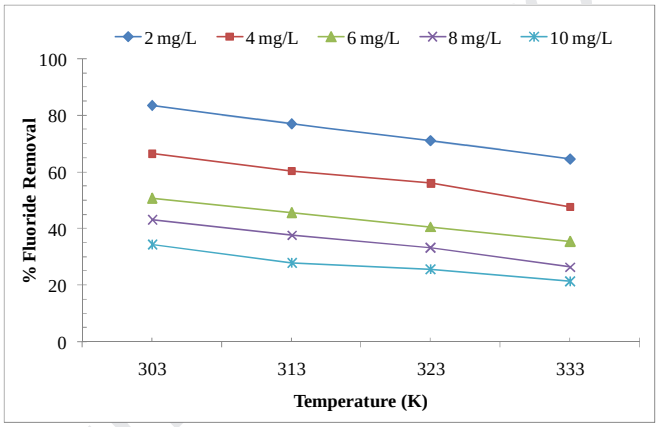

Figure 20: Effect of temperature on fluoride adsorption by MACAIB.

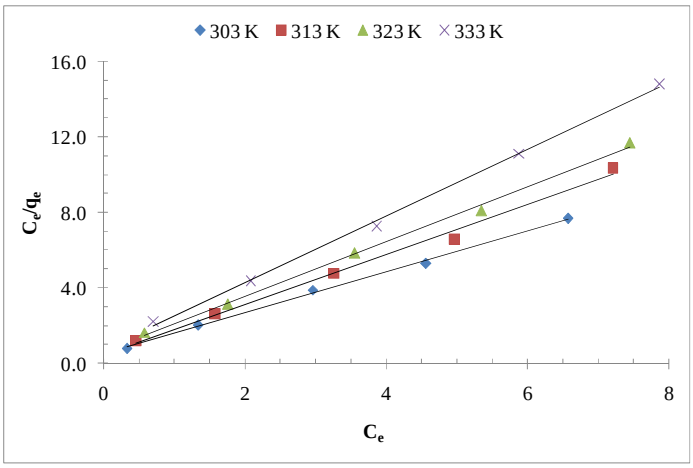

Figure 11: Langmuir isotherm model for adsorption of fluoride by MACAIB.

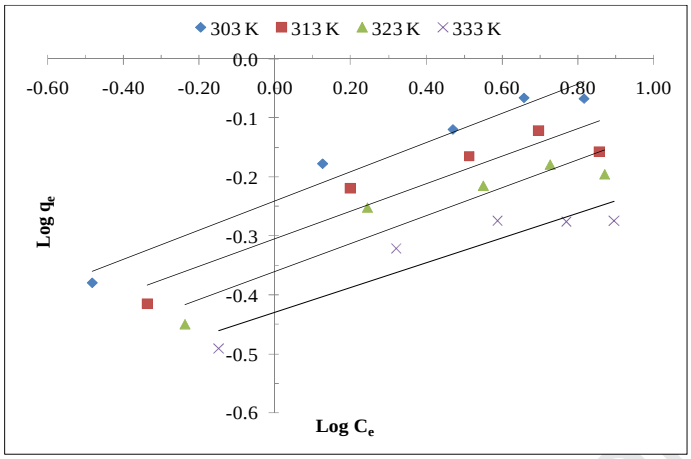

Figure 12: Freundlich isotherm model for adsorption of fluoride by MACAIB.

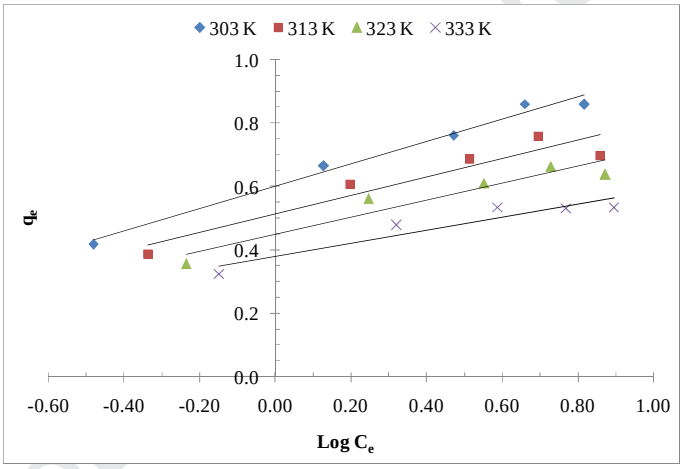

Figure 13: Temkin isotherm model for adsorption of fluoride by MACAIB.

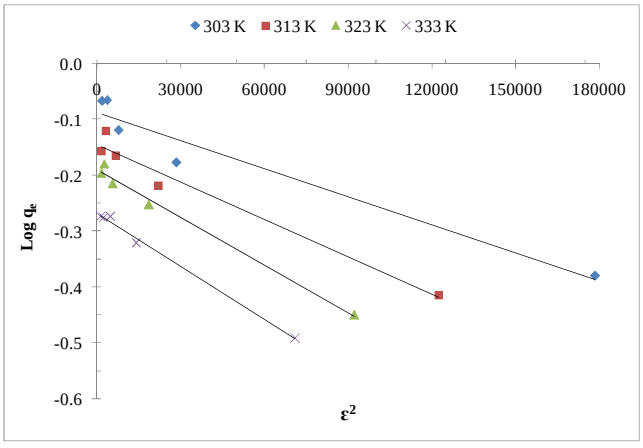

Figure 14: Dubinin-Radushkevich (D-R) isotherm model for adsorption of fluoride by MACAIB.

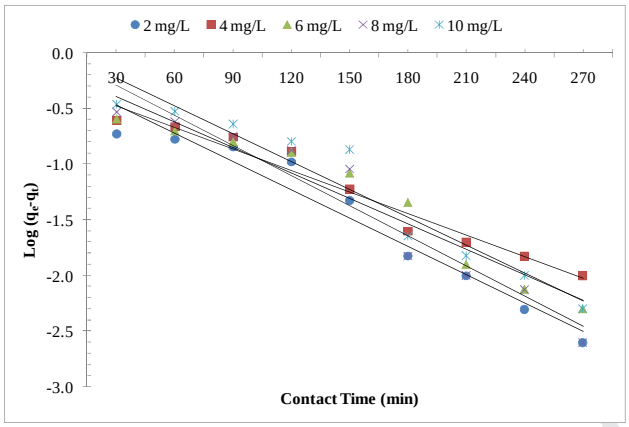

Figure 16 : Plots of Pseudo first-order kinetic model for adsorption of fluoride on MACAIB.

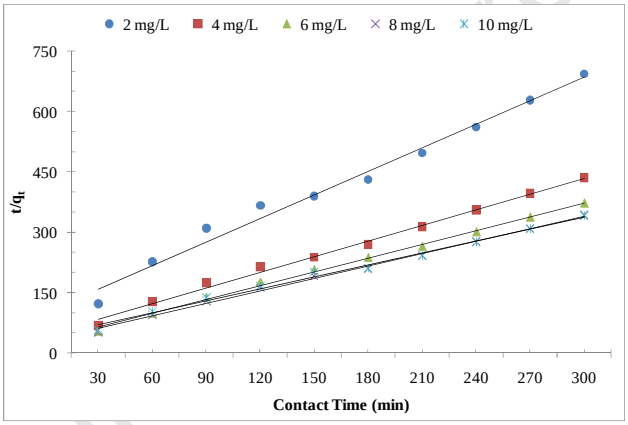

Figure 17: Plots of Pseudo second-order kinetic model for adsorption of fluoride on MACAIB.

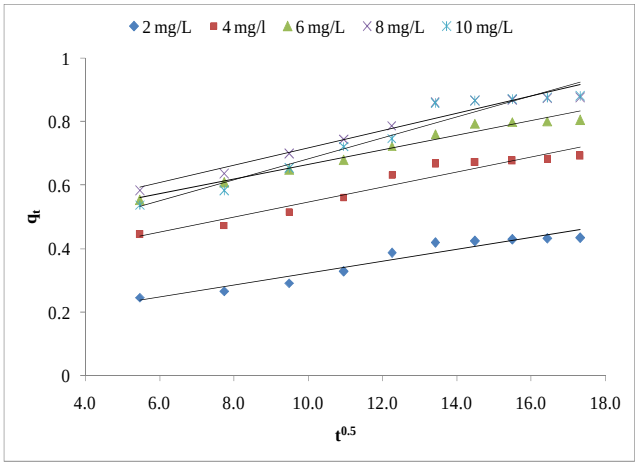

Figure 18 : Plots of Intra-particle diffusion model for adsorption of fluoride on MACAIB.

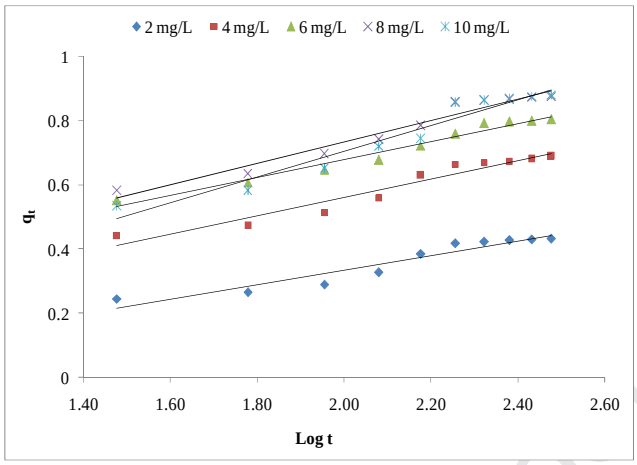

Figure 19: Plots of Elovich kinetic model for adsorption of fluoride on MACAIB.

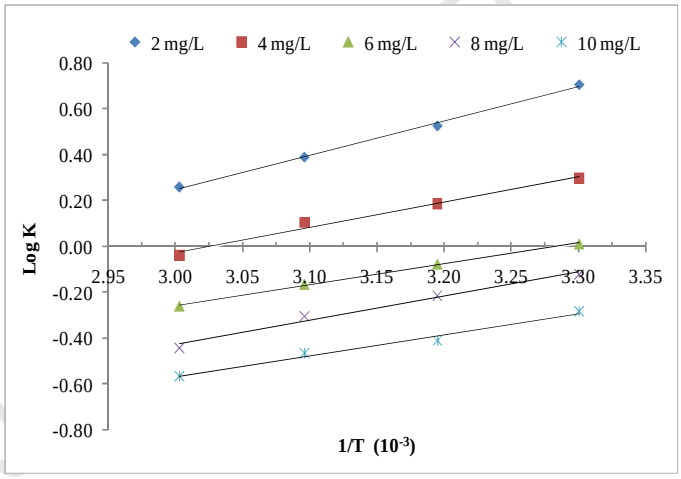

Figure 21: Plots of Log K versus 1/T for adsorption of fluoride by MACAIB.

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Table 1 : Proximate analysis of MACAIB material

S. N.	Parameters	Values
1	Bulk density (gm/cm^3)	0.46
2	Moisture content%	5.73
3	Ash content %	12.85
4	Volatile matter content %	16.44
5	Fixed carbon content %	64.98
6	pH	7.36
7	Water Soluble Matter (%)	0.93
8	Acid soluble matter (%)	3.87

Table 2 : Ultimate analysis of MACAIB material

S. N.	Parameters	Values
1	Carbon %	58.54
2	Hydrogen %	1.48
3	Nitrogen %	1.39
4	Sulphur %	ND
5	Oxygen %	38.59
6	Surface Area (m^2/g)	65.82
7	Average Pore Diameter (A^O)	99.79
8	Total Pore Volume (cc/g)	0.11

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Table 3 : FT-IR absorption bands and possible functional groups of MACAIB before and after fluoride adsorption

IR Peaks	Before adsorption	After adsorption	Difference	Possible functional groups
1	3422.20	3425.13	2.93	-N-H and –O-H stretching
2	2871.63	2917.91	46.28	Aliphatic -C-H asymmetric str.
3	2514.85	2510.99	-3.86	$-CH_2$ symmetric str.
4	2356.70	2358.06	1.36	Aliphatic –C-H asymmetric str.
5	2055.85	Disappeared	--	Aliphatic –C-H symmetric str.
6	1794.56	1805.09	10.53	-C=O stretching
7	1434.85	1438.71	3.86	-N-H bending
8	1049.14	1054.92	5.78	-C-N stretching
9	873.64	873.64	0	-C-O-C-, –C-N stretching
10	717.42	708.39	-9.03	-C-O stretching
11	605.57	576.64	-28.93	-C-C- deformations, -C-H deformations
12	711.64	713.57	1.93	-C-C- stretching.

Table 4 : EDX analysis results of MACAIB before fluoride adsorption.

Element	Weight %	Atomic %
C K	51.10	62.17
O K	35.48	32.60
Mg K	0.46	0.28
Si K	0.29	0.16
P K	0.38	0.18
K K	0.31	0.12
Ca K	11.98	4.49

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Table 5 : EDX analysis results of MACAIB after fluoride adsorption.

Element	Weight %	Atomic %
C K	47.37	56.32
O K	38.82	38.09
F K	0.47	0.39
Na K	0.13	0.09
Mg K	0.47	0.29
Si K	0.28	0.15
P K	0.37	0.19
Ca K	12.09	4.48

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Table 6: Empirical adsorption isotherm and kinetic equations and constant parameters [9]

Isotherm Models	Isotherm Equations and Constant parameters	Kinetic Models	Kinetic equations and constant parameters
Langmuir adsorption isotherm	$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$ (3) $\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m}$ (4) $R_L = \frac{1}{1 + K_L C_0}$ (5)	Pseudo first-order kinetic model	$\frac{dq}{dt} = k_1 + (q_e - q_t)$ (12) $log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right) t$ (13)
Freundlich adsorption isotherm	$q_e = K_F C_e^{1/n}$ (6) $lnq_e = lnK_F + (1/n)\ \ln C_e$ (7)	Pseudo second-order kinetic model	$\frac{dq}{dt} = k_2 (q_e - q_t)^2$ (14) $\left(\frac{t}{q_t}\right) = \left(\frac{1}{k_2 q_e^2}\right) + \left(\frac{1}{q_e}\right) t$ (15)
Temkin Isotherm	$q_e = \frac{RT}{b_T} \ln (A_T C_e)$ $q_e = \frac{RT}{b_T} \ln (A_T) + \frac{RT}{b_T} \ln (C_e)$ $B = \frac{RT}{b_T}$ $q_e = B \ln A_T + B \ln C_e$ (8)	Intra-particle diffusion model	$q_t = k_d t^{1/2} + C$ (16)
Dubinin-Radushkevich isotherm	$lnq_e = lnq_D - (K_D)\ \varepsilon^2$ (9) $E = \frac{1}{\sqrt{2 K_D}}$ (10) $\varepsilon = RT \ln\left(1 + \frac{1}{C_e}\right)$ (11)	Elovich kinetic model	$dq_t = A e^{(-Bqt)}$ (17) $q_t = \left(\frac{1}{B}\right) ln AB + \left(\frac{1}{B}\right) ln t$ (18)

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Table 7 : Adsorption isotherms parameters and coefficients for removal of fluoride on MACAIB

Isotherm	Parameters	Temperature			
		303 K	313 K	323 K	333 K
Langmuir	q_m (mg/g)	0.923	0.755	0.690	0.567
	K_L (L/mg)	2.091	2.880	2.222	2.393
	R_L	0.193	0.148	0.184	0.173
	R^2	0.997	0.993	0.997	0.998
Freundlich	K_F (mg/g)	0.575	0.497	0.438	0.372
	1/n	0.247	0.233	0.235	0.210
	N	4.049	4.292	4.255	4.762
	R^2	0.964	0.887	0.883	0.864
Temkin	K_T (L/mg)	50.316	57.928	48.749	70.596
	B (J/mol)	0.153	0.126	0.116	0.089
	b_T (kJ/mol)	16.481	19.936	21.810	28.301
	R^2	0.982	0.898	0.909	0.884
Dubinin-Radushkevich	q_D (mg/g)	0.817	0.716	0.647	0.540
	K_D (mol^2/kJ^2)	3.84E-06	5.13E-06	6.54E-06	7.28E-06
	E (kJ/mol)	0.361	0.312	0.276	0.262
	R^2	0.948	0.969	0.989	0.995

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Table 8 : Adsorption kinetics parameters and coefficients for removal of fluoride on MACAIB

Kinetic Model	**Parameters**	**Initial fluoride concentrations (mg/L)**				
		2	**4**	**6**	**8**	**10**
Pseudo first-order	q_e (mg/g) [Plot]	0.606	0.526	0.688	0.950	1.044
	q_e (mg/g) [Expt.]	0.432	0.690	0.805	0.875	0.881
	k_1 (min^{-1})	0.583	0.447	0.525	0.622	0.573
	R^2	0.951	0.951	0.936	0.937	0.932
Pseudo second-order	q_e (mg/g) [Plot]	0.424	0.647	0.793	0.868	0.873
	q_e (mg/g) [Expt.]	0.432	0.690	0.805	0.875	0.881
	k_2 (g/mg min)	0.549	0.529	0.512	0.445	0.343
	h (mg/g min) (10^{-1})	0.099	0.222	0.323	0.334	0.250
	R^2	0.984	0.994	0.996	0.996	0.991
Intra-particle diffusion	k_d ($mg/g\ min^{0.5}$)	0.018	0.023	0.023	0.027	0.033
	C	0.134	0.309	0.432	0.441	0.352
	R^2	0.913	0.941	0.971	0.945	0.945
Elovich	A (mg/g min) (10^{-1})	0.295	1.132	3.182	2.243	0.997
	B (g/mg)	10.235	8.024	8.225	6.874	5.743
	R^2	0.918	0.938	0.969	0.957	0.942

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Table 9 : Thermodynamic adsorption parameters for removal of fluoride onto MACAIB

C_0 (mg/L)	ΔH (kJ/mole)	ΔS (kJ/mol K)	ΔG (kJ/mole)			
			303 K	313K	323 K	333 K
2	-28.452	-0.081	-4.085	-3.145	-2.405	-1.653
4	-21.023	-0.064	-1.728	-1.110	-0.648	0.249
6	-17.391	-0.057	-0.067	0.452	1.015	1.653
8	-20.257	-0.069	0.710	1.288	1.887	2.825
10	-17.591	-0.066	1.638	2.471	2.880	3.619

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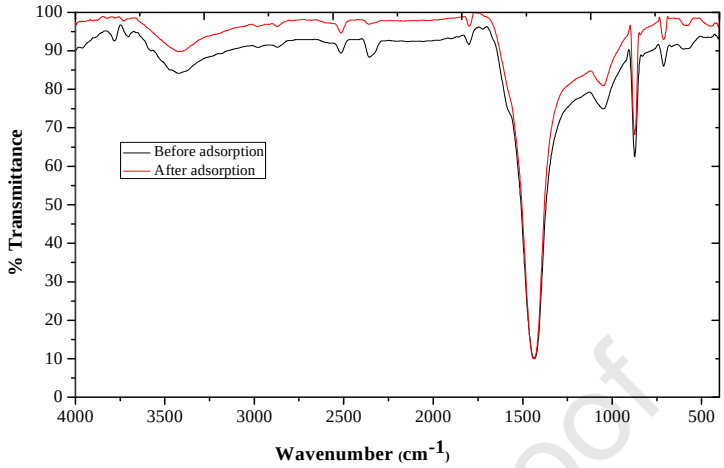
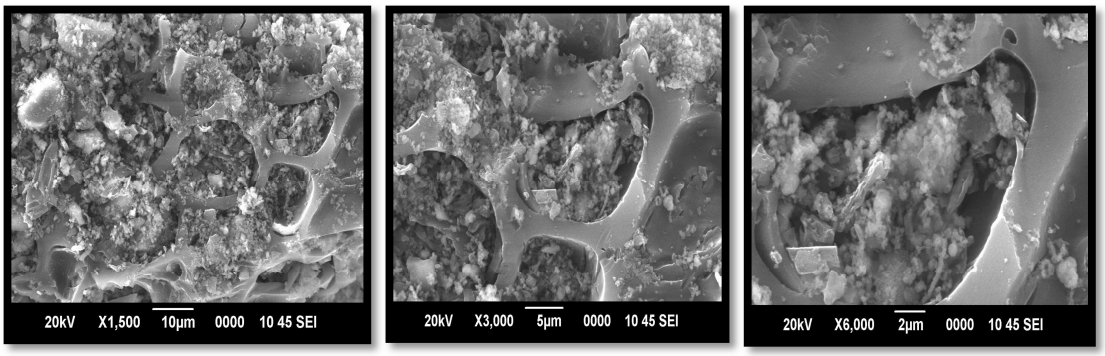
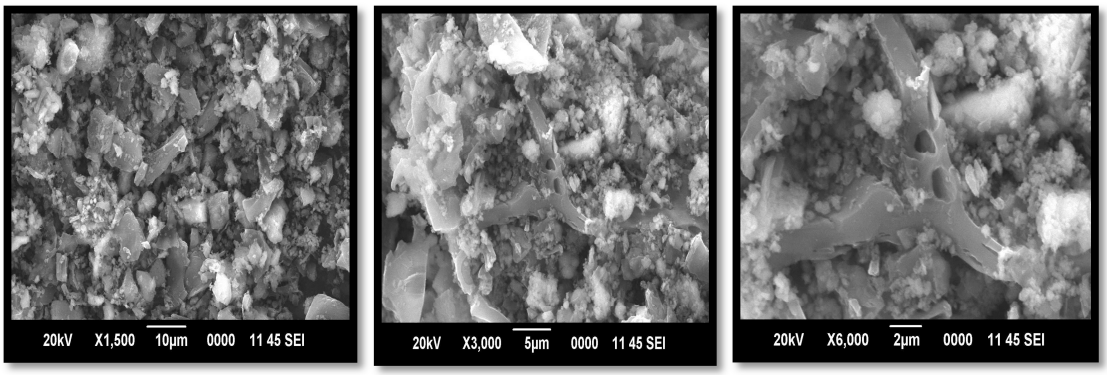


Figure 1 : Fourier Transform Infrared (FTIR) spectra of MACAIB before and after fluoride adsorption.



(a) (b) (c)

Figure 2 (a, b, c) : SEM micrographs (10, 5 and 2 µm) of MACAIB before fluoride adsorption.



(a) (b) (c)

Figure 3 (a, b, c) : SEM micrographs (10, 5 and 2 µm) of MACAIB after fluoride adsorption.

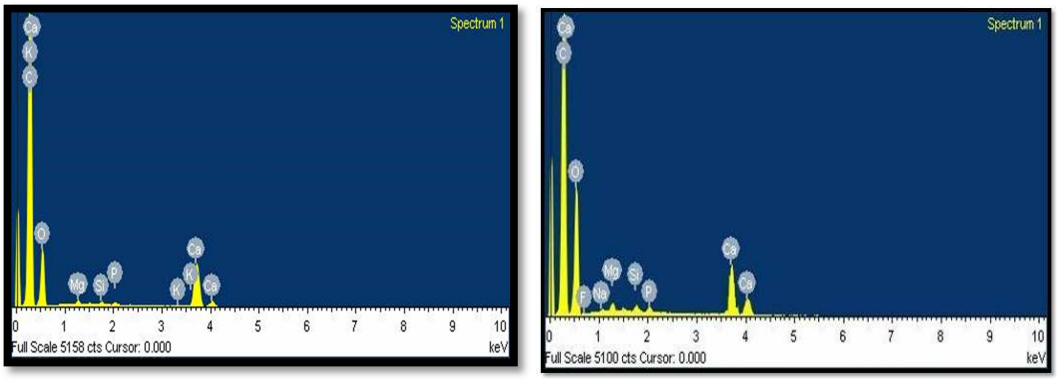


Figure 4 : EDX monograph of MACAIB before and after fluoride adsorption.

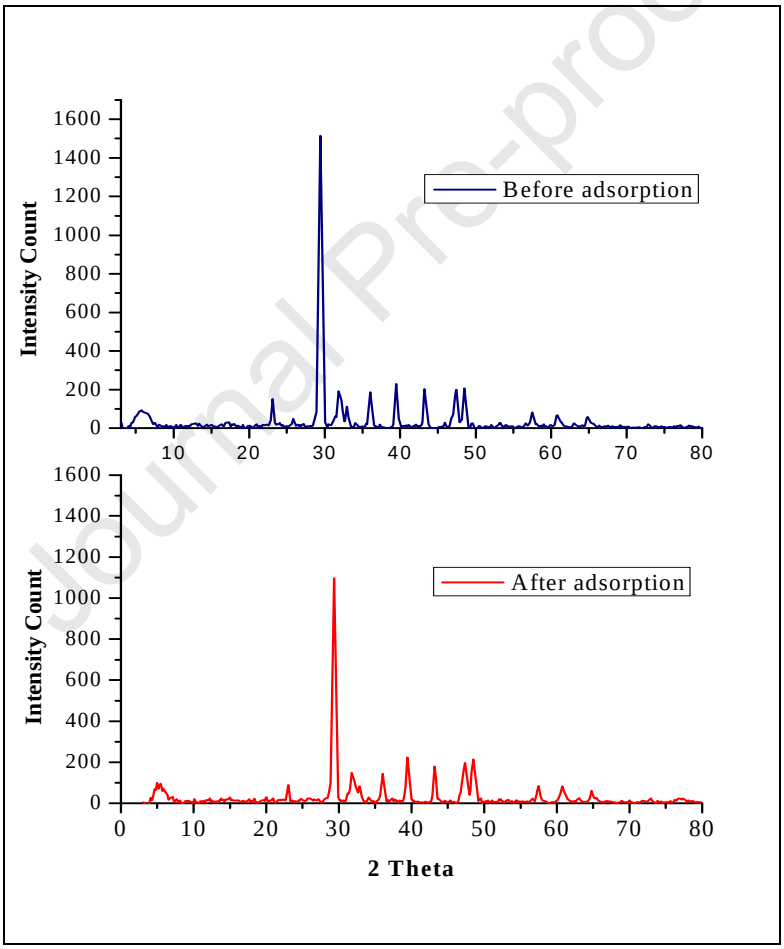


Figure 5 : Powder X-ray diffraction (XRD) of MACAIB before and after fluoride adsorption.

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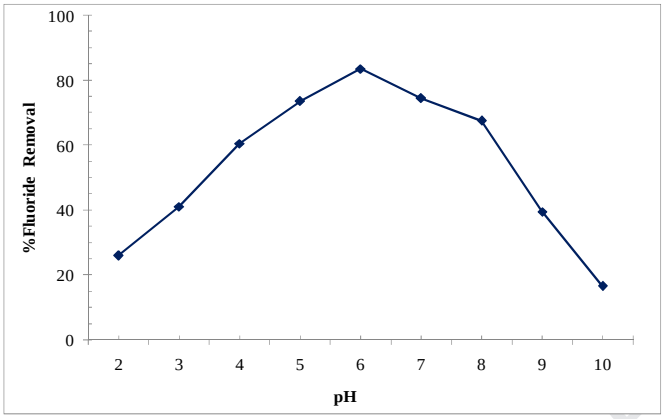

Figure 6: Effect of pH on fluoride adsorption by MACAIB

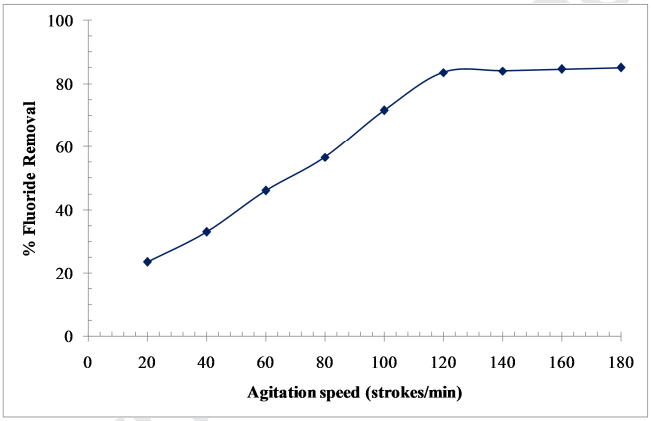

Figure 7: Effect of agitation speed on fluoride adsorption by MACAIB

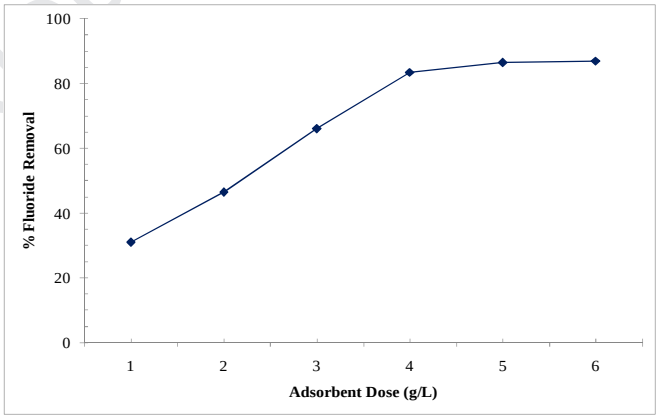

Figure 8: Effect of adsorbent dose of MACAIB on fluoride adsorption.

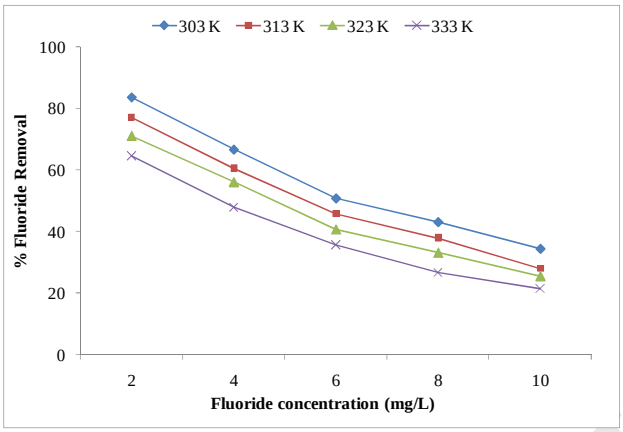

Figure 9: Effect of fluoride concentration on its adsorption by MACAIB.

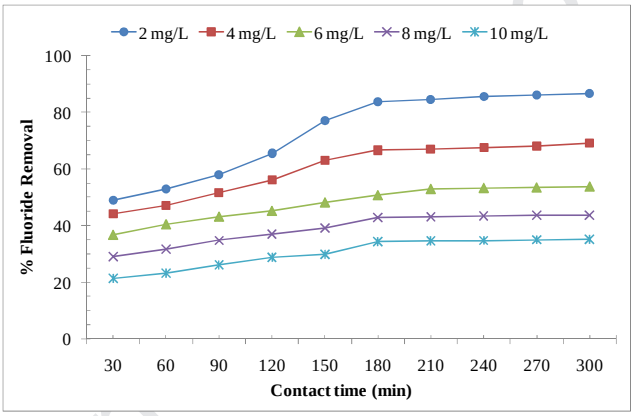

Figure 10: Effect of contact time on fluoride adsorption by MACAIB.

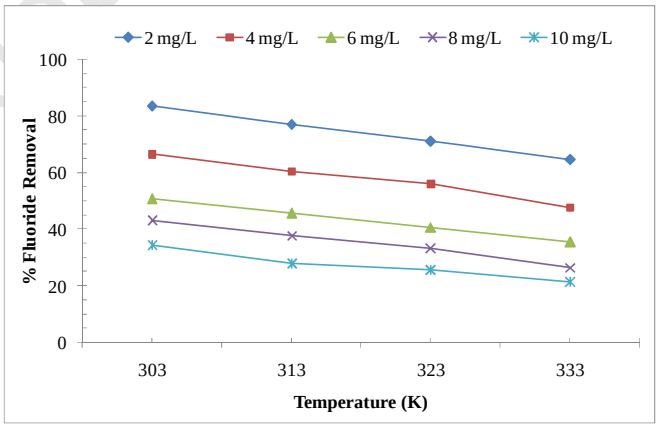

Figure 11: Effect of temperature on fluoride adsorption by MACAIB.

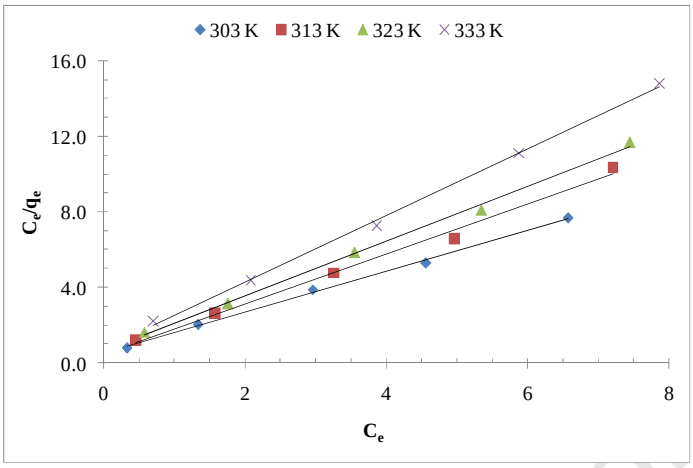

Figure 12: Langmuir isotherm model for adsorption of fluoride by MACAIB.

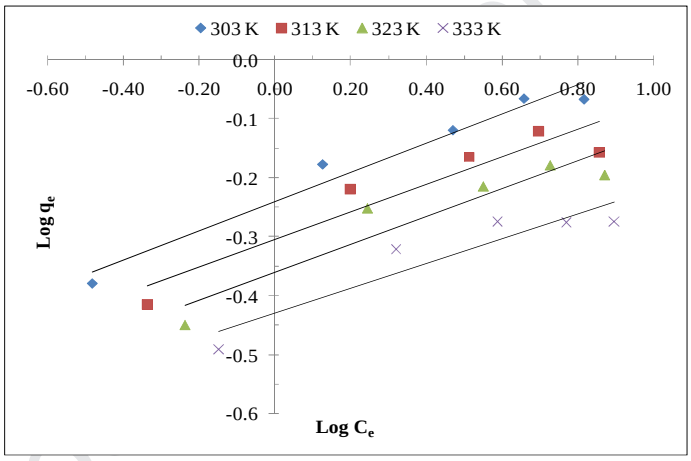

Figure 13: Freundlich isotherm model for adsorption of fluoride by MACAIB.

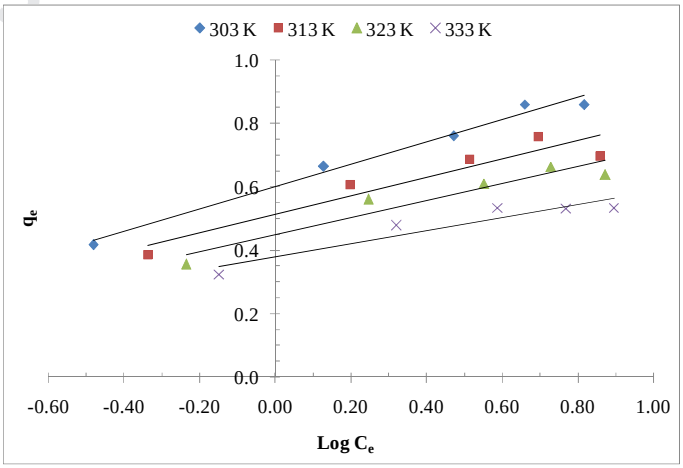

Figure 14: Temkin isotherm model for adsorption of fluoride by MACAIB.

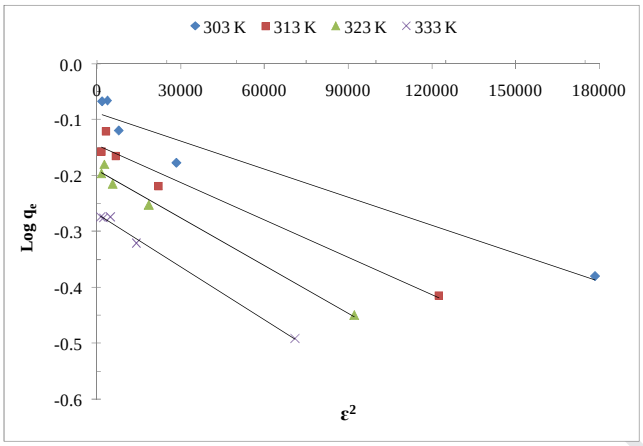

Figure 15: Dubinin-Radushkevich (D-R) isotherm model for adsorption of fluoride by MACAIB.

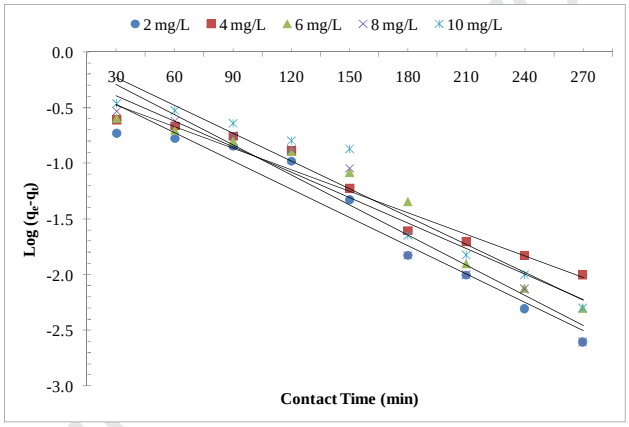

Figure 16 : Plots of Pseudo first-order kinetic model for adsorption of fluoride on MACAIB.

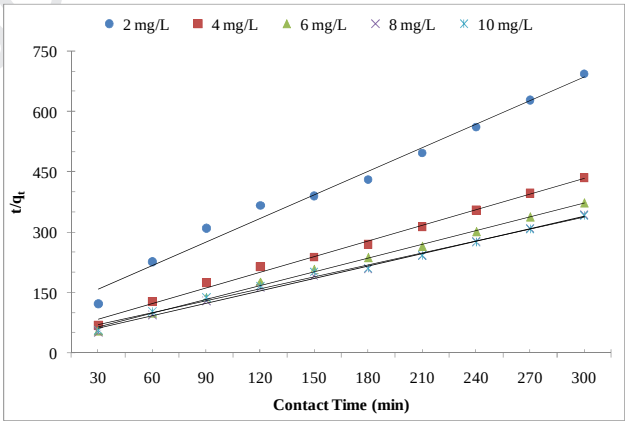

Figure 17: Plots of Pseudo second-order kinetic model for adsorption of fluoride on MACAIB.

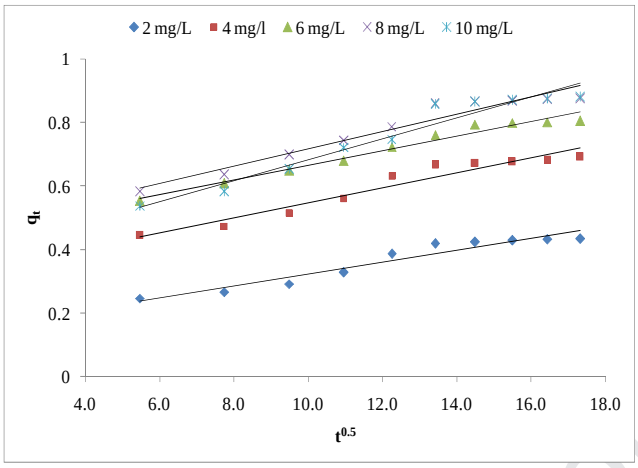

Figure 18 : Plots of Intra-particle diffusion model for adsorption of fluoride on MACAIB.

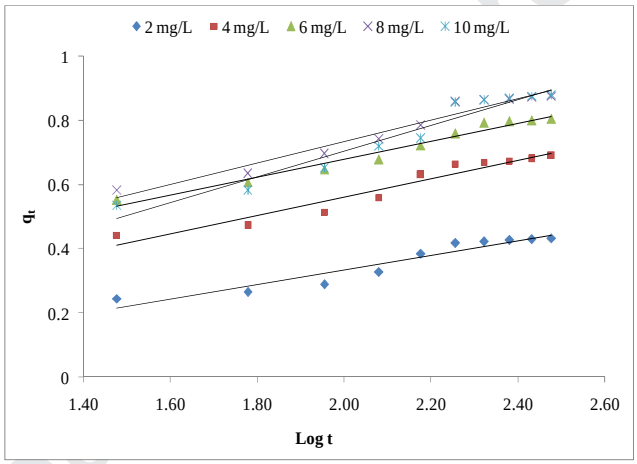

Figure 19: Plots of Elovich kinetic model for adsorption of fluoride on MACAIB.

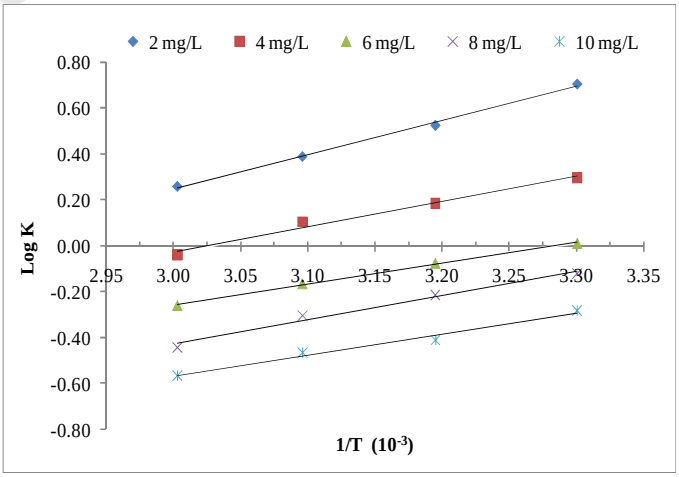

Figure 20: Plots of Log K versus 1/T for adsorption of fluoride by MACAIB.

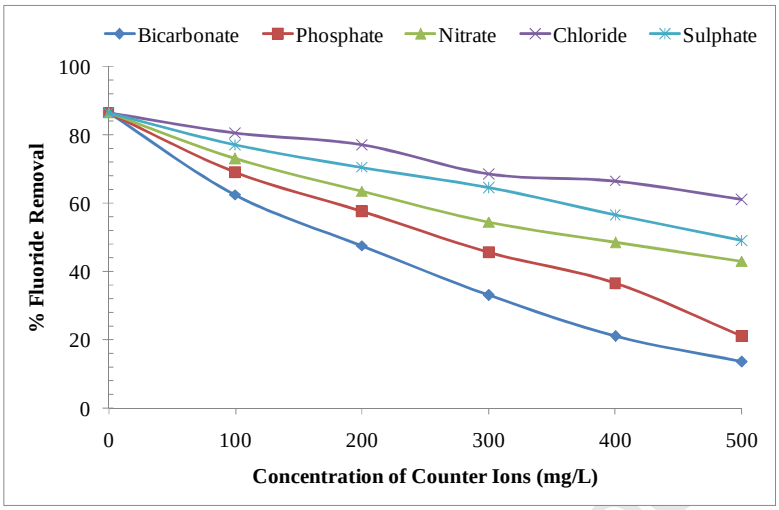

Figure 21: Effect of counter ions on fluoride adsorption by MACAIB.

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