DEFLUORIDATION OF WATER BY ACTIVATED CARBON PREPARED FROM HERBAL PLANTS

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ABSTRACT:

A batch adsorption measure was picked on Activated Carbon produced using locally accessible trees Serianthes-nelsonii, Tridax Procumbence and Cassia auriculata to evaluate their appropriateness for expulsion of fluoride from fluoride sullied water. The productivity of adsorption of fluoride was found to rely upon physical parameters actual boundaries like portion of dose of the adsorbent, pH, contact time, stirring rate, particle size of the adsorbent and initial adsorbate concentration in water. These boundaries were contemplated to discover their ideal qualities for the take-up of fluoride.

3The fluoride evacuation effectiveness in all the three adsorbents considered was observed to be 95.5% (Serianthes-nelsonii), 90% (Tridax Procumbens) and 50% (Cassia auriculata). The ideal adsorbent dosages were observed to be 12 gm/L for Serianthes-nelsonii, 14 gm/L for Tridax Procumbens and 12 gm/L for cassia auriculata. Harmony was accomplished in 100 min for Serianthes-nelsonii, just as for Tridax Procumbens and 80 min for Cassia Auriculata, an upgraded adsorption was acquired at pH 2. It was tracked down that the adsorption of fluoride on these Activated Carbon follows the Langmuir isotherm.

KEYWORDS: Fluoride Removal; Batch Adsorption Techniques; In Expensive Adsorbents; Adsorption Isotherms.

1. INTRODUCTION

In India, at the beginning of the twenty-first century, the rural drinking water supply programme provided a provisional report on groundwater. This report showed a decrease in incidence of water-borne diseases; however, the report also highlighted the diminution of drinking water sources and pathological pollution of the drinking water source. Fluoride is a regular component of natural water. In India, endemic fluorosis is a It is a serious concern in 18 of the 29 states, particularly in Rajasthan, Andhra Pradesh, Tamil Nadu, Gujarat, and Uttar Pradesh, and its concentration fluctuates depending on the source of water (Manisha P et al., 2013).Water has a significant impact on man's daily activities. There are many applications of water in human life which includes drinking, bathing, livestock production, industrial, transportation, cooking, washing, irrigation, recreation and sports, hydroelectric power generation, construction, fishery and agriculture (Simmons I.G et al., 1999). Population growth, industries and agricultural development are major reasons for constantly increasing demands for water. Hence, countries worldwide are endeavouring (striving) to improve the evaluation of their water resources. Drinking water quality, quantity, and availability are among the most pressing environmental, social, and political concerns today's international level (Dahiya S et al., 2007). In addition to other impurities, fluoride is found in natural water in small concentration. India is facing major problem in the form of high fluoride concentration in ground water sources (Agarwal V et al., 1997). One such life-threatening water pollutant is fluoride. Its gaseous form is an extremely potent oxidizer. Fluoride appears in nature as a highly reactive fluoride ion, with a natural abundance of 0.065–0.09% by weight in the Earth's crust. (Mondal et al., 2014).The World Health Organization (WHO) established a safe fluoride level in drinking water at 1–1.5 mg/L. (Swain et al., 2012). Admission of fluoride from drinking water at the degree of 1 mg/L upgrades bone turn of events and forestalls dental conveys. Surpassing the restriction of fluoride admission might prompt dental and skeletal fluorosis. The primary constituent of teeth and bone is hydroxyl apatite (Ca10 (PO4)6(OH) 2) and fluoride can substitute the hydroxide particle inside the hydroxyl apatite gem construction to frame fluorapatite (Ca_5

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$(PO_4)_3F$), which makes teeth and bones denser, harder, and more brittle (Yu X et al., 2013). Young children are the most susceptible as dental enamel and skeletal formation is most active during early childhood. Moreover, excessive fluoride intact may interfere in DNA synthesis Table 1: Effects of concentration of Fluoride.

C_f (mg/L)	Effect
< 1.0	Reduces dental caries
1.0 – 1.5	Marginal risk of fluorosis
1.5 – 3.0	High risk of dental fluorosis
3.0 – 10.0	Leads to skeletal fluorosis with adverse change in bones
>10.0	Crippling and skeletal fluorosis

Therefore, researchers are trying to focus on various defluoridation processes. The most commonly used methods for defluoridation of drinking water are reverse osmosis, precipitation, membrane filtration, ion-exchange, etc.Among these, adsorption is considered as the best method for its low maintenance cost and effectiveness towards fluoride removal even at low concentrations (Gandhi et al., 2012). Hence, researchers have focused on locally available bio-adsorbents like Moringa oleifera (drum stick), tulsi, tamarind seed, tea ash (Mondal et al., 2014), babool bark,banana peel (Bhaumik & Mandal, 2015), neem leaf, kikar leaf (Kumar et al., 2008), papal leaf , red mud, fly ash, rice husk ash, and maize ash.

Henceforth, in this work, we looked for dynamic carbons got from plant materials having a place with various classes of plant realm. Our essential examinations demonstrated that there is solid partiality among fluoride and dynamic carbons got from leaves Serianthes-nelsonii, Tridax Procumbence and Cassia Auriculata plant. Along these lines, this work is given to contemplate inside and out the sorption qualities of the said dynamic carbon towards fluoride as for different physicochemical boundaries like adsorbent measurements, molecule size, temperature, contact time, impact of pH, and beginning fluoride fixation and Characterisation of the enacted carbon is dissected by utilizing Fourier change infrared spectroscopy (FTIR) and X-beam diffraction (XRD) investigation.

2. MATERIAL AND METHODS

Of the various classes of plant materials tested for their sorption abilities towards fluoride, it has been noted that the Activated Carbon derived from the leaves of Serianthes-nelsonii, Tridax Procumbens and Cassia-Auriculata plant shows affinity for fluoride. The plant were collected surrounding areas of JNTUA Anatapuram.

2.1Preparation of Fluoride stock solution

The stock solution of 100 mg/L fluoride was prepared by dissolving 221 mg of anhydrous NaF in 1000 mL of double distilled water, such that each ml of solution contains 1 mg of NaF. The samples are prepared at different concentrations of 2, 4, 6,8,10 and 12 mg/L are ready by weakening of the stock arrangement. All safeguards are required to limit the misfortune because of dissipation during the readiness of arrangements and ensuing estimations.

2.2 Preparation of Activated Carbon

The Herbal plants of Serianthes-nelsonii, Tridax Procumbence and Cassia auriculata were collected from a site around JNTUA. The plant leaves were separated from the plant, double distilled water were used remove dirt and other particulate matter. The leaves were air dried for 48 hours and the dried leaves were then crushed separately in a grinder to get a fine powder. The Activated carbon of three plants were prepared separately by treating the leaves powder with the concentrated sulphuric acid (Sp. gr.1.84) in a weight ratio of 1:1.8 (biomaterial: acid). The resulting black product is kept in a Muffle furnace, maintained at 400°C for 4 h followed by washing with double distilled water until free of excess acid, then dried at 400°C for 30 minutes (Singanan M et.al., 2015).The prepared activated carbon is used as adsorbent for the removal of fluoride.

2.3 Experimental Procedure

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Adsorption studies were performed by batch method to obtain the optimum value of parameters. All the steps of the experiment were performed in 250 ml conical flasks at room temperature. Standard fluoride ion solution (mg/L) was prepared from sodium fluoride (NaF) and distilled water. Experiments were carried out by adding different ratios of 2g to14g of Activated Carbon in 30 ml fluoride stock solution to study the extent of adsorption of fluoride on Activated Carbon. The contents of all the flasks were simultaneously agitated for a particular time on Orbital shaking machine. After stirring, the contents of all the flasks were allowed to settle for two minutes and the supernatants were carefully decanted and filtered through Wattman filter paper No. 42. The filtered solution of the flasks were analysed for residual fluoride ion concentration by using HACH Pocket Colorimeter.

3. RESULTS AND DISCUSSIONS

The impact of different boundaries like adsorbent measurement, pH, contact time, Stirring Rate, beginning fluoride particle focus and molecule size which influence the expulsion of fluoride particles were improved by fluctuating every one of them at a time by keeping the excess boundaries steady. The outcomes are introduced underneath.

3.1 Effect of Adsorbent Dose

To study the effect of an increase in the dose of adsorbent on the fluoride removal efficiency, the experiment was conducted with increasing doses of Activated Carbon ranging from 2 gm/L to 14 gm/L. The pH of the solution was adjusted to the required level, using (0.1N) HCl or NaOH solutions and the initial fluoride ion concentration was fixed 2 mg/L. The contents of all the flasks containing 30 ml (2 mg/L standard fluoride) solution were simultaneously mixed at 130 rpm for 120 min in an Orbital shaker. The effect of adsorbent dose on fluoride removal is shown in Fig 3.1.

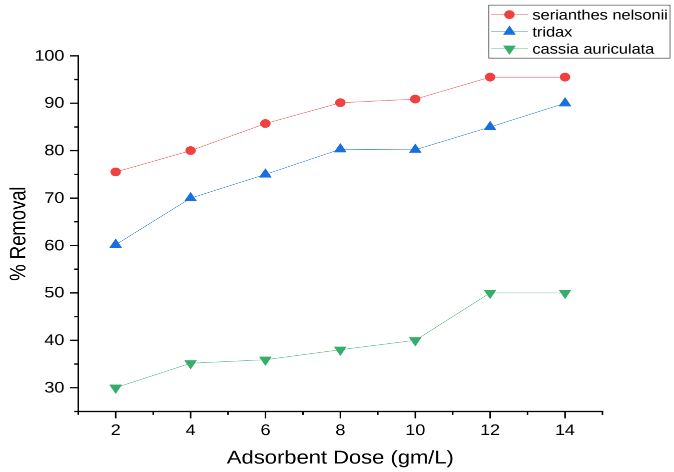

Fig3.1: Effect of Adsorbent Dose on Fluoride Removal

(Initial F- concentration = 2 mg/L; contact time =100 min for Serianthes nelsonii and Tridax Procumbens, 80 min for Cassia Auriculata; pH = 2; stirring rate = 110 rpm for Serianthes nelsonii and Tridax Procumbens and 130 rpm for Cassia Auriculata).

It is observed that the adsorption of fluoride increases with the increase in the amount of adsorbent dose. This might be due to the fact that with higher doses of adsorbent more pore volume and larger surface area would be available to the adsorbate. Hence higher is the removal of fluoride ions. It is also observed that the removal of fluoride increases with the dose but after a certain dose range; there is no significant increase in removal (Srimurali M et.al., 1998). The result shows that Activated Carbon of Serianthes nelsonii removes 75.5% of fluoride with a dose of 2 gm/L which increases to 95.5% with 12 gm/L dose of adsorbent Tridax Procumbens achieves 60.16% and 90% fluoride removal at doses of 2 gm/L and 14 gm/L and Cassia Auriculata achieves 30 % and 50 % fluoride removal at doses of 2 gm/L and 12 gm/L respectively, at room temperature of 26 ± 0.5^{0}C. There is less significant removal after a dose of 12 gm/L for Serianthes nelsonii, 14 gm/L for Tridax Procumbens and 12 gm/L for Cassia Auriculata. Hence the respective doses were selected for the further studies.

3.2 Effect of PH

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To study the influence of pH on removal of fluoride test mixture containing 2.0 mg/L of F^- and optimum dose were studied on various pH values i.e. 2, 4, 6 and 8. The pH of the solution containing fluoride ion was adjusted by adding (0.1N) HCl or NaOH solutions. As the pH of the test fluoride solution increases from 2.0 to 8.0, the fluoride removal reduces from 95% to 76.1% for Serianthes nelsonii, 90% to 60% for Tridax Procumbens and from 50% to 20% for Cassia Auriculata. Graphically the results of the experiments are shown in Fig 3.2.

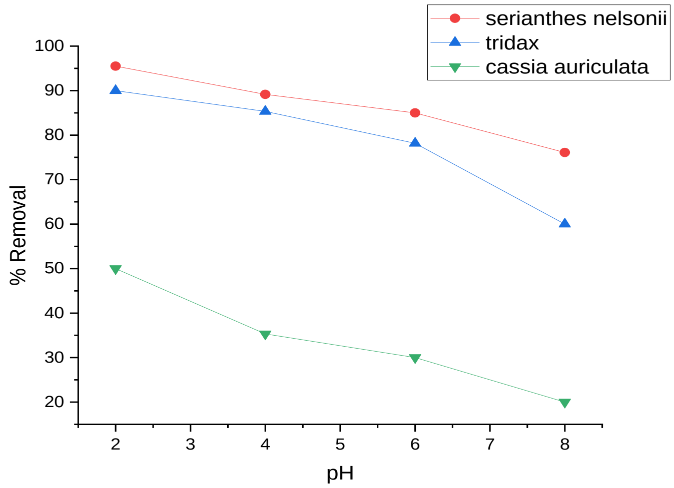

Fig.3.2: Effect of pH on Fluoride Removal

(Initial F^- concentration = 2 mg/L; contact time = 100 min for Serianthes nelsonii and Tridax Procumbens, 90 min for Cassia Auriculata; Adsorbent dose = 12 gm/L for Serianthes nelsonii, 14 gm/L for Tridax Procumbens and 12 gm/L for Cassia Auriculata; stirring rate = 110 rpm for Serianthes nelsonii and Tridax Procumbens and 130 rpm for Cassia Auriculata)

Thus it concluded that removal of fluoride ions decreases with the increase in pH of the solution. The reason for the better adsorption at low pH might be due to large number of H^+ ions present at these pH values which in turn neutralizes the negatively charged OH^- ions present on the adsorbent surface thereby reducing hindrance to the diffusion of fluoride ions (Srimurali M et.al., 1998). Thus pH 2 was taken for further study.

3.3 Effect of Contact time

The study of effect of contact time was done with the standard fluoride solution of 2 mg/L and an optimum dose of adsorbent was added at optimum pH value. The contact time of these mixtures were varied from 20 min to 120 min. The result of the study is shown in Fig 3.3.

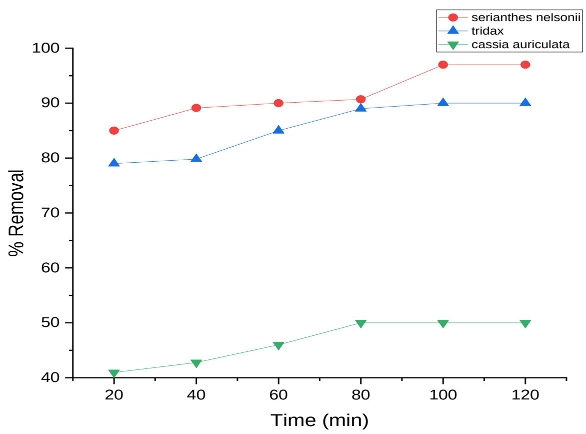

Fig.3.3 Effect of Contact time on Fluoride Removal

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(Initial F- concentration = 2 mg/L; pH = 2; Adsorbent dose = 12 gm/L for Serianthes nelsonii, 14 gm/L for Tridax Procumbens and 12 gm/L for Cassia Auriculata; stirring rate = 110 rpm for Serianthes nelsonii and Tridax Procumbens and 130 rpm for Cassia Auriculata).

It is evident that adsorption of fluoride increases with increase in contact time and after some time it can also be seen that equilibrium was established at about 100 min. But further increase of contact time did not increase the uptake of fluoride by the adsorbent. This might be due to non-availability of the adsorption sites on the adsorbent surface [Jamode A.V et.al, 2004]. The fluoride uptake increases from 85% to 97% for Serianthes nelsonii, 79% to 90% for Tridax Procumbens and 40.95% to 50% for Cassia Auriculata at 20 min to 120 min of contact time respectively. Equilibrium time was obtained at 100 min for Serianthes nelsonii and Tridax Procumbens. Equilibrium time at 80 min was obtained for Cassia Auriculata.

3.4 Effect of Stirring Rate

The effect of stirring rate was studied by varying the agitation speed from 20 rpm to 140 rpm at optimum pH with optimum doses of all the three adsorbents with optimum contact time. The effect of stirring rate on fluoride removal is shown in Fig3.4.

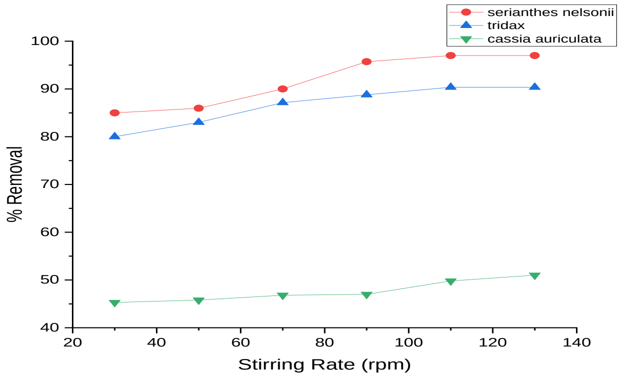

Fig.3.4: Effect of Stirring Rate on Fluoride Removal

(Initial F- concentration = 2 mg/L; pH = 2; Adsorbent dose = 12 gm/L for Serianthes nelsonii , 14 gm/L for Tridax Procumbens and 12 gm/L for Cassia Auriculata; contact time = 100 min for Serianthes nelsonii and Tridax Procumbens and 80 min for Cassia Auriculata)

Fluoride removal depends upon the stirring rate. An increase in adsorption was observed from 85% to 97% for Serianthes nelsonii, 80% to 90.35% for Tridax Procumbens and 45.3% to 51% Cassia Auriculata for when the stirring rate was increased from 30 rpm to 130 rpm. The percentage adsorption is less at lower stirring rate and increases with the stirring rate up to 110 rpm for Serianthes nelsonii and Tridax Procumbens. The adsorption increases up to 130 rpm in case of Cassia Auriculata and thereafter remains almost constant. Due to stirring, proper contact was developed between F^- ion solution and the binding sites, which promoted effective transfer of adsorbate ions on to the adsorbent sites (Hem J.D et al., 1989).

3.5 Effect of Initial Concentration

Studies on the effect of initial fluoride ion concentration was conducted by varying the initial fluoride ion concentration of the standard fluoride solution from 2 mg/L to 12 mg/L at optimized set of conditions. The result of analysis is shown in Fig 3.5.

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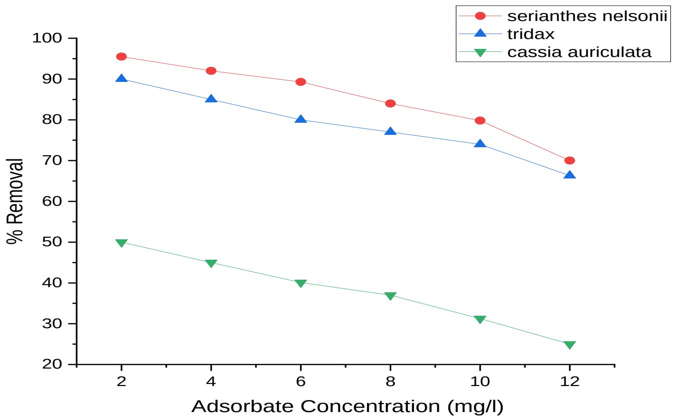

Fig3.5: Effect of Initial Concentration on Fluoride Removal

(Initial F- concentration = 2 mg/L; pH = 2; Adsorbent dose = 12 gm/L for Serianthes nelsonii, 14 gm/L for Tridax Procumbens and 12 g/L for Cassia Auriculata; contact time = 100 min for Serianthes nelsonii and Tridax Procumbens and 80 min for Cassia Auriculata; stirring rate = 110 rpm for Serianthes nelsonii and Tridax Procumbens and 130 rpm for Cassia Auriculata)

The removal of fluoride ion decreases with increase in initial fluoride concentration. The adsorption capacity of Initiated Carbon was found to increment with decline in introductory fluoride particle focus. This was presumably because of the way that for a decent adsorbent portion, the complete accessible adsorption destinations were restricted, consequently adsorbing practically a similar measure of fluoride and accordingly, showing a diminishing in rate evacuation of fluoride relating to an expanded beginning fluoride particle fixation (Hem J.D et al., 1989) and (USEPA et.al., 1996).

3.6 Effect of Particle Size

The experiment was conducted to evaluate the influence of adsorbent particle size on the fluoride removal. The experiment was done with fluoride ion solution of 2 mg/L, pH 2, and optimum time of contact, optimum dose of adsorbents and with stirring rate. The results are shown in Fig 3.6.

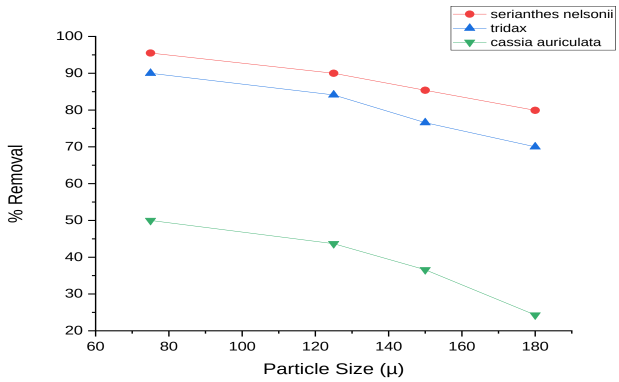

Fig.3.6 Effect of Particle Size on Fluoride Removal

(Initial F- concentration = 2 mg/L; pH = 2; Adsorbent dose = 12 gm/L for Serianthes nelsonii, 14 gm/L for Tridax Procumbens and 12 gm/L for Cassia Auriculata; contact time = 100 min for Serianthes nelsonii and Tridax Procumbens and 80 min for Cassia Auriculata; stirring rate = 110 rpm for Serianthes nelsonii and Tridax Procumbens and 130 rpm for Cassia Auriculata)

The removal of fluoride increases as the particle size decreased from 180 µ to 75 µ. The fluoride removal efficiency decreases from 95% to 79.93% for Serianthes nelsonii, 90% to 70% for Tridax Procumbens and 50% to 24.3% for Cassia Auriculata. The relatively higher adsorption rate as the particle size decreases due to increase in more active surface area (USEPA et.al., 1996).

3.7 FTIR spectroscopy of Activated Carbon

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It gives confirmation for the presence of specific functional groups on the surface of carbon materials. Several characteristic bands were observed in the FTIR spectrum of Activated Carbon Prepared from Serianthes nelsonii, is shown in Fig.8 shows the surface functional groups with peaks at O-H group 3484.61cm^{-1}, C-H group 2929.09cm^{-1}, C=O group 1720.8cm^{-1}, C=C group 1632.91cm^{-1}, C-O group 1184.49cm^{-1} presence of carboxyl groups attributed to Activated carbon (Nageswara Rao et al. 2011)

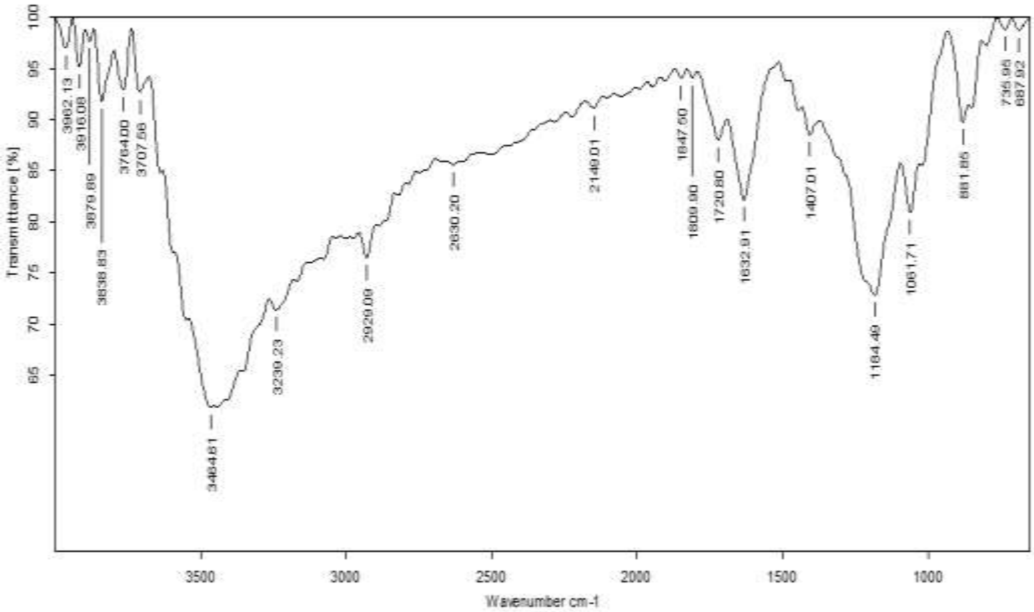

Fig.3.7 FTIR spectrum of Activated carbon

3.8 X-Ray Diffraction Analysis

X-ray diffraction analysis is used to find the crystalline or the amorphous nature of the synthesized activated carbon. From the obtained XRD pattern, the activated carbon made from Serianthes-nelsonii plant leaves showed the main peaks at 2θ values of 21.51° and 21.44° as shown in the fig 15.The crystalline size of the activated carbon particles was estimated to be from the Debye Scherrer formula (ChityalGanesh et.al., 2016).

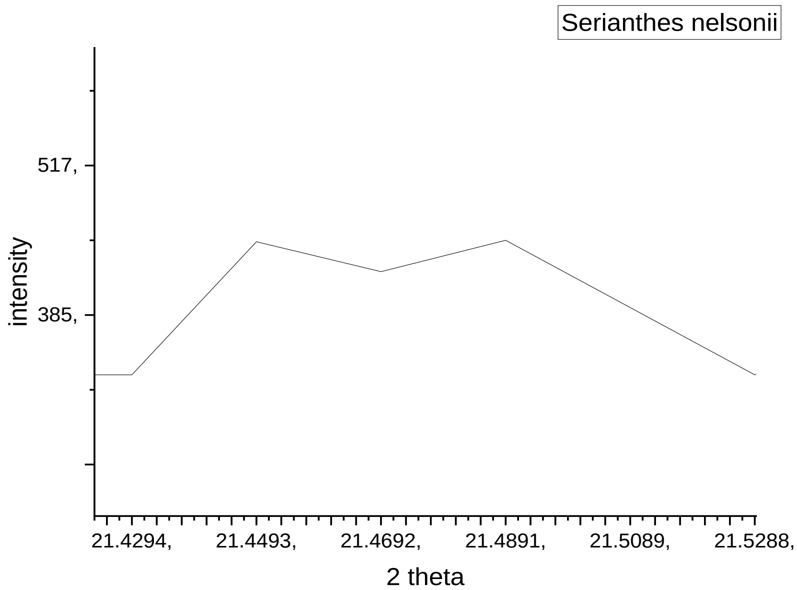

Fig.3.8 X-Ray Diffraction pattern of Activated carbon (Serianthes-nelsonii)

4. ADSORPTION ISOTHERM

The adsorption isotherm is only a numerical connection between the sum adsorbed on the adsorbent surface and the tension (if there should be an occurrence of vaporous adsorbate) or the fixation (in the event of fluid) of the

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adsorbate in harmony at a consistent temperature. To portray the adsorbate-adsorbent co-operations quantitatively and to concentrate on the viability of the permeable for the specific adsorbate, the adsorption information of the adsorbents for the expulsion of fluoride particles have been corresponded with Freundlich and Langmuir isotherm.

4.1 Langmuir isotherm

Langmuir isotherm is legitimate for single layer or monolayer adsorption. It depends with the understanding that all the adsorption destinations have equivalent partiality for atoms of the adsorbate and there is no immigration of adsorbate in the plane of the Surface. The Langmuir equation is commonly written as,

$$\mathbf{q_e = q_{max} K_L C_e / (1+K_L C_e)}$$

The linear form of Langmuir isotherm can be expressed as,

$$\mathbf{1/q_e = (1/q_{max}) + (1/K_L q_{max} Ce)}$$

Where, $\mathbf{q_e}$ is the amount of solute adsorbent per gram of adsorbent (mg/g).

$\mathbf{C_e}$ is the equilibrium adsorbate concentration in solution, mg/L.

$\mathbf{q_{max}}$ is the number of moles of solute adsorbed per unit weight of adsorbent in forming a monolayer on the surface.

$\mathbf{K_L}$ is a constant related to energy. Langmuir isotherm was plotted with $\mathbf{1/q_e}$ against $\mathbf{1/C_e}$ as shown in Fig 4.1, 4.1.1 and 4.1.2.

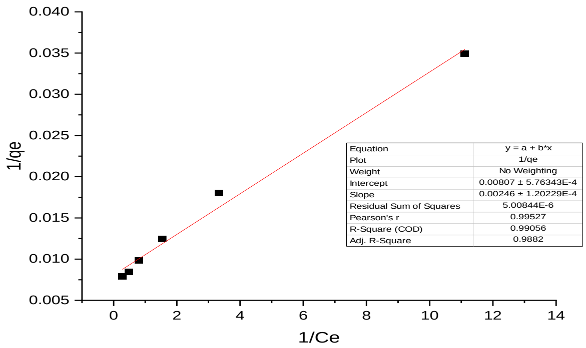

Fig.4.1 Langmuir adsorption isotherm for Serianthes nelsonii

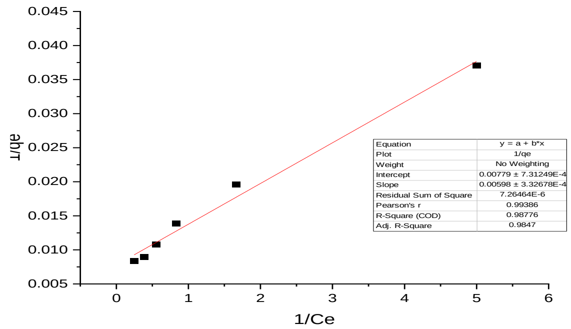

Fig.4.1.1 Langmuir adsorption isotherm for Tridax Procumbens

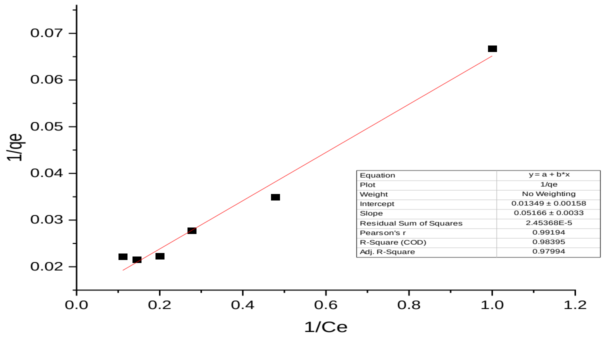

Fig.4.1.2 Langmuir adsorption isotherm for Cassia Auriculata

4.2 Freundlich Isotherm

The Freundlich isotherm is for the most part material to Physico – synthetic adsorption on heterogeneous surface energy frameworks (non-uniform dispersion of adsorption heat). The linear form of the Freundlich adsorption isotherm is represented as,

$$\mathbf{q_e = K_f C_e^{1/n}}$$

The linearized Freundlich adsorption isotherm is given by equation

$$\mathbf{\log (q_e) = \log(K_f) + 1/n \log C_e}$$

$\mathbf{q_e}$ is the amount of adsorbate adsorbed per unit weight of adsorbents, mg/g.

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C_e is the equilibrium adsorbate concentration in solution, mg/L.
K_f and 1/n are the Freundlich constants.
Freundlich isotherm was obtained by plotting with **log(q_e)** against **log(C_e)** as shown in Fig 4.2 ,4.2.1 and 4.2.2.

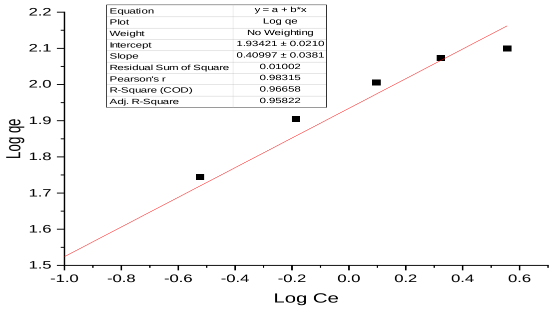

Fig.4.2 Freundlich adsorption isotherm for Serianthes nelsonii

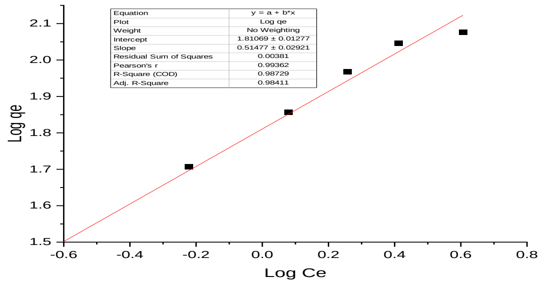

Fig.4.2.1 Freundlich adsorption isotherm for Tridax Procumbens

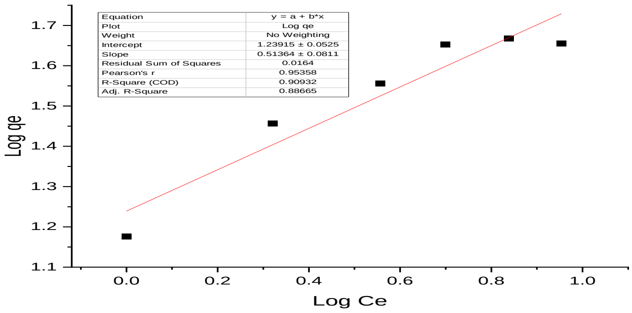

Fig 4.2.2 Freundlich adsorption isotherm for Cassia Auriculata

It could be seen that the value of R^2 for Serianthes-nelsonii, Tridax Procumbence and Cassia auriculata is 0.9882, 0.9847 and 0.9799 hence the process of defluoridation using Activated Carbon of all these three trees follows the Langmuir isotherm well. Thus it shows that the fluoride efficiency by Activated Carbon of Serianthes-nelsonii is more than Tridax Procumbence; which in turn more than Cassia auriculata.

5. CONCLUSION

The results of the experiments show that these low cost and easily available adsorbents could be successfully used for the removal of fluoride from the water over a wide range of concentrations. The percentage of fluoride removal is found to be a function of adsorbent dose, pH, contact time, stirring rate, particle size of adsorbent and the initial adsorbate concentration in water. The process of adsorption follows Langmuir isotherm. The exhausted Activated Carbon can be disposed of safely after use by burning. Thus these bio-adsorbents could be suitably utilized for the treatment of fluoride bearing water on domestic or community levels especially in rural areas.

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