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Synthesis and Characterization of Zinc Oxide Nanoparticles from *Solanum Nigrum* and *Eclipta prostrata* for Effective Removal of Fluoride

M Kavisri[a*], Marykutty Abraham[b], & Meivelu Moovendhan[c]

[a]School of Building and Environment, Department of Civil Engineering, Sathyabama Institute of Science and Technology, Chennai 600 119, India

[b] Sathyabama Institute of Science and Technology, Center for Remote sensing and Geoinformatics, Chennai 600 119, India

[c]Centre for Ocean Research, Sathyabama Institute of Science and Technology, Chennai 600 119, India

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The present study reports the novel approach to remove the excess fluoride in water using nano sized zinc oxide (ZnO) particles extracted from the plant leaf *Solanum nigrum* and *Eclipta prostrate*. The thermal behavior of the fabricated ZnO particles were characterized by XRD, the porosity and microstructure were studied by the SEM. The measurement of FTIR was carried out to find the probable biomolecules in both plant leaves. These leaves contain high amount of proteins, amino acids and rich in polyphenols. The batch experiment was carried out to study the removal of Fluoride under several factors like the effects of initial concentration, adsorbent dosage and various time intervals. It is revealed at optimum dosage of *Eclipta* and *Solanum* is 0.8g/100ml and the capacity of adsorption was creating to be 1.984mg/g and 1.943 mg/g and its efficiency is 98% and 55% respectively. Thus, the optimum dosage of the adsorbents can be used to lowering the fluoride concentration. The synthesized ZnO Nanoparticles in this work are highly capable material for removal of Fluoride ion. Thus, it is proven that the leaf of Solanum nigrum and *Eclipta prostrata* produce higher efficiency in reduction of Fluoride.

Keywords: Eclipta prostrate, Solanum nigrum, Zinc oxide, Adsorption, Nano particles

1 Introduction

In small concentration, fluoride is measured an essential composite for human health. Exposure to high attentions of fluoride present in the groundwater is the main reason for a thoughtful disease namelyfluorosis[1]. Clean water is one of the most important elements for all living organisms to sustain life[2]. The classified of the major contaminants in water for human consumption is mainly the fluoride by the World Health Organization (WHO), in adding to nitrate and arsenic, which cause large-scale health problems[3]. The existence of fluoride in intake water has giving some health assistances for consumers such as dipping dental cavities, but an extreme consumption of this anion or its attendance at high absorption (> 1.5 mg/L) can lead to dental problems and Undernourished Fluorosis[4]. There are few reports representing that fluoride may affect with DNA synthesis. The additional absorptions of fluoride can also inhibit with lipids, vitamins, proteins, carbohydrate and vitamin metabolism[5]. In addition, fluoride (F-) has been revealed to toxic kidney functioning at high doses over short-term contacts in both wildlife and human beings[4,5]. It was also concluded by some research groups that fluoride has the capability to inhibit with the purposes of the mind and pineal gland[6]. Pineal gland problem is a main site of fluoride gathering inside the body, with higher absorptions of fluoride than either bone orteeth problem[5,6]. Fluoride contact has also been related to cancer in urinary bladder generally among workers showing to extra fluoride in the workplace[5]. Hence, the removal of fluoride from water and waste waters is very important before hand these are mixed up with clean natural water resources. Conventional methods such as, nanofiltration technique, electrocoagulation technique, ion exchange removal technique, reverse osmosis technique, electrodialysis technique, precipitation technique and membrane separation technique have been useful for fluoride removal from water and waste waters[7,8]. Some of the Chemical coagulants alike Aluminum sulphate(alum), Ferrous Chlorideis used in the Municipal intake water treatment plant for the decontamination process[6]. An additional use of an amount of chemical coagulants can affect human health, e.g., Al has also been showed to be a causative agent in neurological

*Corresponding author:
(E-mail: kavisrimanikannan1985@gmail.com)

diseases such as pre-senile dementia[6]. Adsorption methods using natural adsorbents or cultivated waste products are becoming the new alternatives for the removal of fluoride from aqueous solution as they are low-priced, modest, sludge-free, regenerable, eco-friendly, involve small initial cost, and minimal chemical use[6,9]. In recent years the adsorbents were coated with other elementsto advance the efficiency removal of fluoride such as zirconium impregnated collagen fiber[10] ceramic coating with iron oxide and alumina Fe-Al ceramic coating etc[8-10]. Several techniques including precipitation, phytoremediation (adsorption), ion exchange and membrane have been widely studied for the removal of additionalquantities of fluoride in water[11]. Among them, adsorption process is commonly accepted due to its cost-effectiveness and comfort in operation[11-13]. Calcium peroxide (CaO_2) is a low-cost material and it has the advantages of a bigger surface area, which raises the reaction rapidity and higher adsorption rate. Though, several studies have specified that the adding of calcium peroxide in ground water is a suitable choice for pollutant degradation, Silica Nanoparticles was derived from rice husk in capacitated on calcium peroxide ($SiCaO_2$) might be an actual adsorbent for defluoridation[14].

Like various adsorbents has widely used to eliminate fluoride such as charcoal[15,16] alumina, alum saturated activated alumina, red mud, bone char (contains tricalcium phosphate or hydroxyapatite 57–80%)[17-19], dried *Crocus sativus* leaves[20], Co-polymeric resin[21],chitosan coated bentonite[22], hybrid adsorbent is fictitious with Mg/Fe composite oxide and alginate[23], Ultra-fine MgO nanoparticles (3–7 nm)[24]. Various materials which occurs naturally and it is available abundantly free cost of charge have been explored as adsorbents for the removal of fluoride from water[12]. Phytoremediation of fluoride using *Nerium oleander*, *Pogonatherumcrinitum*, and *Portulaca oleracea* were observed to remove fluoride up to 92, 80, and 73%, respectively, from NaF solution at the concentration of 10 mg L^{-1} (Tukey-Kramer multiple comparison Test)[25], *Phyllanthus emblica* as a natural adsorbent (enriched in calcium ions) potentially paved a way improving the forms of adsorption[26], white rot fungus (*Pleurotuseryngii*) [27]. The *Solanum nigrum* is belongs to Solanaceae family. It is represented by 53 species. It is used in DNA Sequence and chemical composition and it is used in large scale synthesis of nanoparticles and acts as a reducing agent. It used in the different application like antibacterial and microbial activities against isolated human pathogen and medicinal properties, rich in antioxidants, irons vitamins, minerals and it is rich in calcium to build a strong bond[28,29]. *Ecliptaprostrata*is belongs to daisy family. It is used as a traditional medicine that grows in moist areas. It prevents anemia and a liver diseases and skin related problem. It is helpful to treat hepatitis infection, jaundice in liver cirrhosis, body aching and knees weaken and Joints weaken, blood spitting, epistaxis, diarrhoea with bloody, haematuria and uterine bleeding[30,31]. In this study the green method[26,28] using *Solanum nigrum and Eclipta prostrata* plant leaves has been used as a reducing material at first time as well as used as a surface stabilizing agent of the synthesis of round shaped ZnO nano particles. The ZnO nano materials can be used as a Nano-optical and Nano-electrical devices in industry, and in food packaging and used as a medicine as antimicrobial agents[32-37] and antitumor agents[19,25,29]. Nanoparticles are motivating for cancer application and it have multi-task helping simultaneously as therapeutic mediators, clinical diagnostics and drug distribution mediators[36-38]. The growing focus on Nano zinc oxide resulted in the expansion and development methods of synthesis of Nano particles[32]. Hence, in this work, the phase, structure and morphology of produced product[38,39] were examined by the normal description techniques and to study in detail on the interaction of zinc oxide nanoparticles with fluoride[40,41], the outcome of various parameters viz. contact time, initial fluoride. Further, the methodologies adoptability advanced in this work has been tested with diverse groundwater samples were collected from 5 different districts in Tamilnadu[37].

2 Materials and Methods

2.1 Plant extracts preparation

In this study, *Solanum nigrum* and *Eclipta prostrata* leaves were washed methodically with distilled water[15]. About 10g of leaves were taken and chopped nicely[28]. Then add 100ml of distilled water which were boiled at 100º Celsius for 10 min[40]. Then, the extract was filter with filter paper (What man No.1)[15,26] twice and filtrate was collected in 100ml beaker. This extract was then directly used in the synthesis of ZnO Nanoparticles as shown in Figure 1.

2.2 Synthesis of Zinc oxide Nano particles

In this method, 1.36g of zinc chloride was dissolved in 100ml of water. In this 100ml of water

Table 1 — Water quality analysis

Locations	Fluoride (Mg/L)	Iron (Mg/L)	Chloride (Mg/L)	Nitrate (Mg/L)	Sulphate (Mg/L)	pH	TDS (Mg/L)	Hardness (Mg/L)	Alkalinity (Mg/L)
Tiruchengode	2.2	0.7	579.36	1.04	29.84	8.1	2418	481	1080.3
Dindugal	1.53	1.3	8.3	0.79	13.95	6.2	1	130.3	1203.1
Salem	1.033	0.327	198.44	30.428	58.944	6.66	1.3	130.5	120.3
Namakkal	1.1	0.5	729.36	1.25	13.95	7.9	1700	690	675
Kollimalai	0.51	0.3	732	0.93	27.90	8.0	670	889	690

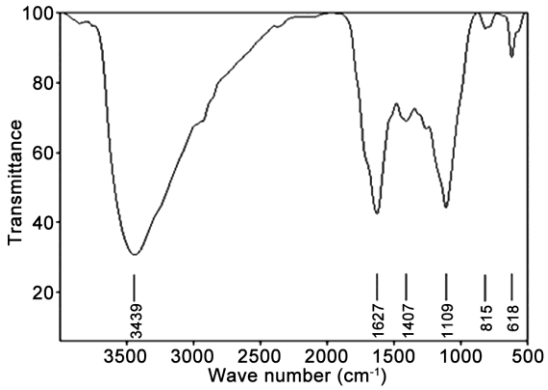

Fig. 1 — FTIR analysis of samples of *Ecliptaprostrata* ZnO Nanoparticles.

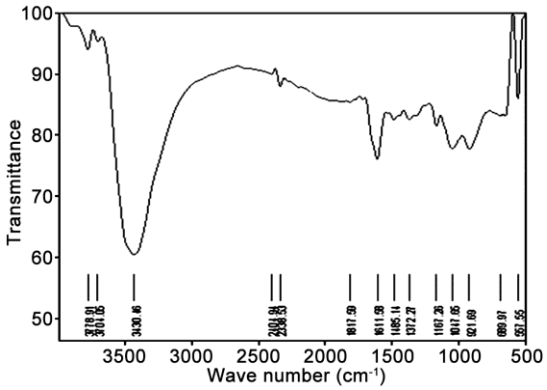

Fig. 2 — FTIR analysis of samples*Solanum nigrum* ZnO Nanoparticles.

50ml has been taken for *Solanum nigrum* and another 50ml for *Eclipta prostrata.* In this solution 50 ml of was taken and kept in magnetic stirrer for 10 min and add the plant extract in the concentration of 10ml, 20ml, 30ml and leave it for 30 min. 0.39 g of sodium chloride was added and kept it 10 min in the temperature of 80^0c and left it for 1 day and the very next day the precipitate will be settled at the bottom.

2.3 Preparation of Nano powder

The precipitate has been placed in the Petri plate kept in the oven for 80^oC for 1 day after that the powder has been kept in the muffle furnaces for 4h on 400^oC[15] and then the powder has been grinded nicely. The same procedure has been adopted for the *Ecliptaprostrata*.

2.4 Analyzing methods of Fluoride

The following are the two methods are employed in this study for analysing the fluoride parameter in water.

2.5 Fluoride testing kit method (color compatibility method)

In this method 5ml of water has been taken in the 10 ml of beaker by using measuring jar. In the 5ml of water, 5 drops of Fluoride reagents were added and dissolving thoroughly. In the presence of fluoride gives a light color[31].

2.6 Experimental study

The sample was collected from different districts such as Tiruchengode, Namakkal, Salem, Dindugal and Kollimalai. The Samples were collected on the basics of total population present in the district according to the guidelines for drinking water quality. Each districts 5 number of samples were collected. The Fluoride level in water from different district was analysed by Tamilnadu water board as shown in Table 1.

2.7 Preparation of standard fluoride solution

Batch mode adsorption studies were adopted by standard protocol[36]. Fluoride stock solution was prepared by 2.21g of sodium fluoride is dissolving in 500 ml of water and the required standard preparation of diluted stock solution for the batch experiment was prepared[42]. The batch experiment is efficiency in the fluoride removal with different dosage of ZnO NPs[33] and has effect in the initial concentration of fluoride of optimum dosage of ZnO NPs. 100ml of fluoride solution has been taken, 800mg of ZnO was added and kept it in the magnetic stirrer for 2 hours and 2 ml of solution was taken in the interval of 10,20,30,40,50,60,90 and 120 mins and the removal of efficiency was studied using optimal dose of absorbent. Preparation of ppm stock solution in the concentration of 1, 0.75, 0.50, 0.25 and 0.1 was given in Fig. 2.

- 1ppm - 25ml of stock solution+25ml of distilled water
- 0.75ppm -18.75ml+31.25ml of distilled water

- 0.50ppm - 12.5ml+37.5ml of distilled water
- 0.25ppm - 6.25ml+43.75mlof distilled water
- 0.1ppm - 2.5ml +47.5ml of distilled water

2.8 Centrifugation of Standard solution

To identity the efficient and effective concentration of Nano powder the first trail was carried out using Nano powder of *Solanum nigrum* and *Eclipta prostrata*. The standard fluoride solution has been treated with ZnO particles with various timing such as 10, 20, 30, 40, 50, 60, 90,120 mins. The 50ml of 2 ppm stock solution +300mg of Nano powder (*Solanum nigrum*) and 800 mg of (*Eclipta prostrata*). The solution has been kept in the magnetic stirrer for 2 hours. 2 ml of solution has been taken in the time interval of 10,20,30,40,50,60,90 and 120 minutes. It has been kept in the centrifuge for 20 min in 3000 rpm.

2.9 Adsorption Calculation

The adsorption capacity had been found using adsorption isotherm equation was done by standard method[2,36,36,42]

$$q_i = \frac{(c_0 - c_t)}{m} v \quad \ldots (1)$$

Where,

q_i - adsorption capacity(mg/g),
Co - Initial concentration (mg/l),
C_t - concentration at time t (mg/l),
V - Volume (L) of water and
m – mass of the absorbance (g)

The efficiency is calculated for all the absorbent using the equation[34,42]. When the fluoride concentration reduces fluoride water, the efficiency of absorbent tends to be more[36,42].

$$\text{Efficiency} = \left[\frac{f_i - f_f}{f_i}\right] * 100 \quad \ldots (2)$$

Where,

f_i – initial concentration of fluoride (mg/l),
f_f – final concentration of fluoride (mg/l),
Generally expressed in terms of percentage (%).

2.10 IR spectra, XRD and SEM analysis

The FT-IR, XRD and SEM analysis was performed for *Eclipta prostrata* and *Solanum nigrum* synthesized nanoparticles by following the standard procedure[36, 42].

3 Results and Discussion

3.1 FTIR Analysis

The measurement of FTIR was carried out to classify the possible bimolecular in *Eclipta prostrata* leaf extract[30,31] and it is responsible for covering leading to effective stabilization of the ZnO nanoparticles[15] and the peaks were obtained at 3439 (O-H), 1627,1407, 1109 and 618 cm^{-1}as shown in Fig. 1. The FTIR spectral analysis was done after the formation of ZnO nanoparticles. The shifted peaks from 3439 to 3486 and 1627 to 1669 cm^{-1}, new peaks appeared at 2922, 2854 cm^{-1} are attributed to the asymmetric stretching vibrations and symmetric stretching vibrations group of $-CH^2$ respectively[33]. A peak was formed at 556 cm^{-1} due to the correspondence of alkyl halides stabilizes the ZnONPs[28]. The region between 400 cm^{-1} and 600 cm^{-1} is allotted for metal-oxygen bond as shown in Fig. 2. The FTIR spectrum of the *Solanum nigrum* showed characteristic peaks at 3414, 1589, 1398, 1092 and 618 cm^{-1} (alkyl halides)[29] (Fig. 2). The FTIR spectra after the formation of ZnO NPs and the shifted peaks from 3414 to 3430 cm^{-1}, disappeared peaks were 1589 and 1398 as shown in (Figs. 1 and 2). New peaks appeared at 2404, 2338, 1817, 1817 and 1611cm^{-1} were reducing the NPs. A peak was formed at 557 cm^{-1} due to the correspondence of alkyl halides stabilizes the ZnO NPs as shown in Figs. 1 and 2.

3.2 XRD Analysis

The result of room temperature X-ray diffraction pattern (XRD) of the prepared samples is shown in Figs 3 and 4. The sharp and most intense peaks observed here indicate that the synthesized ZnO nanoparticles are of high crystalline nature with single phase. The strong diffraction peaks appear at 31.9°, 33.3°, 36.3°, 47.2°, 56.3°, 62.9°, 66.2°, 67.9°, 69.2° and 76.7° corresponding to (101), (001), (102), (103), (110), (104), (200), (113), (200) and (201) the structure of ZnO crystal as shown in (Figs. 3 and 4). The Figs 3 and 4 shows the highest peaks to reduce

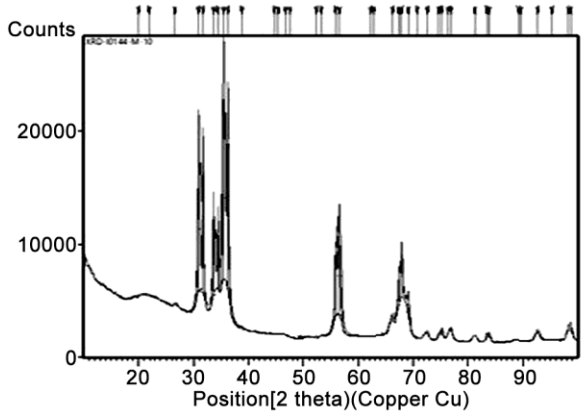

Fig. 3 — XRD analysis of ZnO nanoparticles of *Ecliptaprostrat*

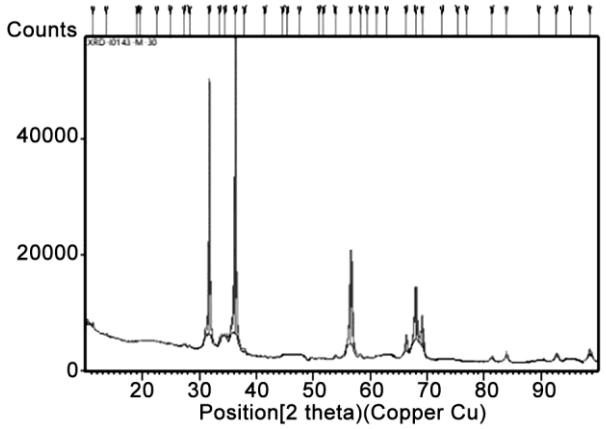

Fig. 4 — XRD analysis of ZnOnanoparticles of *Solanum nigrum*

the fluoride concentration. The Fig. 4 has shown the highest peaks among the Figs 3 and 4 concentrations. The *Solanum nigrum* has an effective one to reduce the fluoride concentration (Fig. 4).

3.3 SEM (Scanning Electron Microscope) analysis

SEM is the most adaptable instruments obtainable for the investigation and study morphology microstructure and characterization of chemical composition[36,40]. SEM image of prepared ZnO NPs[28] were analysed in order to measure the size of the nanoparticles and their surface distribution was presented in Fig. 5.

3.4 Fluoride Absorption Study

3.4.1 Fluoride Adsorption Study in solution

Fluoride solution is an artificial sample prepared at any concentration for the experimental study[23,26]. The solution was prepared with an initial concentration of 50mg/l. This is done to obtain maximum efficiency and adsorption capacity of fluoride and also optimum time and optimum dosage[23]. In order to experiment with the sample water collected in different districts. The concentration of fluoride in standard solutions were analysed by UV spectroscopy[34,36]. In the uv graph, the fluoride has been treated with ZnO NPs for the minimum and optimum dosage of 0.3 g/ml and 0.8 g/ml for the optimum time[20]of 120 min . The first trial was carried out with the concentraion of standard solution in which the fluoride content has been reduced with the optimum time of 120 min as shown in Table 2. The Optimum dosage of *Solanum nigrum and Eclipta prostrata* is 0.8g/100ml and adsorption capacity of *Eclipta prostrata*is found to be 1.984mg/g and its efficiency is 98%. Adsorption capacity of S*olanum nigrum*is found to be 1.943mg/g and its efficiency is 55%. Thus, the optimum dosage of the

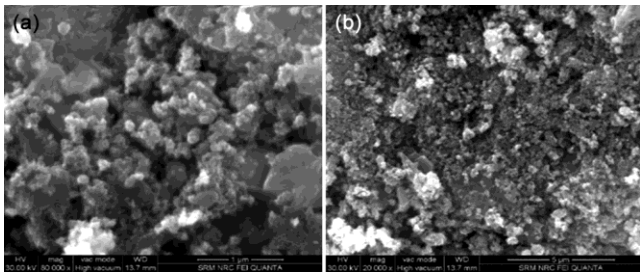

Fig. 5 — SEM analysis of nanoparticles (a) *Solanum nigrum*, (b) *Eclipta prostrate.*

Table 2 ─ Synthesized ZnO nanoparticles treated NaF aqueous solution

Nanoparticles concentrations (mg/L^{-1})		Efficiency (%)
*Ecliptaprostrata*ZnO nanoparticles	300	62.5
	800	98.75
*Solanum nigrum*ZnO nanoparticles	300	49
	800	55

Table 3 ─ Synthesized ZnO nanoparticles treated Tiruchengode ground water

Nanoparticles concentrations (mg)		Efficiency (%)
Eclipta prostrate ZnO nanoparticles	300	50
	800	50
Solanum nigrum ZnO nanoparticles	300	55
	800	97

adsorbents can be used to lowering the fluoride concentration.[34,37].

3.4.2 Fluoride absorption study in ground water

The sample was collected from different districts such as Salem, Namakkal, Tiruchengode, Dindugal, and Kollimalai. Among the districts, Tiruchengode and Namakkal has considered to be the higher concentration of fluoride content. Sample is tested with both physical and chemical parameters[42] such as PH, TDS, Fluoride concentration and adsorption capacity. The samples are tested using *Solanum nigrum* and *Eclipta prostrata* synthesized Nano particles at a minimum dosage of 0.3g and optimum dosage of 0.8g with an optimum contact time of 120 min. The first trail has been carried out by the fluoride standard solution. Among the districts the Tiruchengode and Namakkal area has higher fluoride concentration. In which the fluoride content has been reduced by using the ZnO NPs. The Tiruchengode sample has been tested with the ZnO NPs of *Solanum nigrum*and *Eclipta prostrata* with 300 mg and 800 mg at optimum dosage with the contact time of 120 min. From Table 3, it is revealed at optimum dosage of *Solanum nigrum* and *Eclipta prostrata*is 0.8g/100ml

Table 4 — Synthesized ZnO nanoparticles treated Namakkal ground water

Nanoparticles concentrations (mg)		Efficiency (%)
Eclipta prostrata ZnO nanoparticles	300	83.33
	800	86.66
Solanum nigrum ZnO nanoparticles	300	86.66
	800	93.33

and adsorption capacity of *Eclipta prostrata* was found to be 0.062 mg/g and its efficiency is 50%. Adsorption capacity of *Solanum nigrum* was found to be 1.943 mg/g and its efficiency is 97%. Thus, the optimum dosage of the adsorbents[34,37]can be used to lowering the fluoride concentration. The Namakkal sample has been tested with the ZnO NPs of *Solanum nigrum* and *Eclipta prostrata* of 300 mg and 800 mg at optimum dosage with the variation of contact time. From Table 4, it is revealed at optimum dosage of *Solanum nigrum* and *Eclipta prostrata* is 0.8g/100ml and adsorption capacity of *Eclipta prostrata* was found to be 0.062mg/g and its efficiency is 50%. Adsorption capacity of S*olanum nigrum* was found to be 1.943mg/g and its efficiency is 97%. Thus, the optimum dosage of the adsorbents can be used to lowering the fluoride concentration[43].

4 Conclusion

This study brings out the ill effects of excessive fluoride that affects human health. To overcome these harmful effects, the study has introduced the defluoridation techniques using bio degradable absorbents. As the study is dealt with the waste materials from household, it is said to be a suitable low-cost technique. From the analysis of the experimental study, the absorbent capacity of *Solanum nigrum* and *Eclipta prostrata* is 0.3 mg, 0.8 mg respectively. Thus, it is proven that the leaf of *Solanum nigrum* and *Eclipta prostrata* produce higher efficiency in reduction of fluoride. The sample collected from Namakkal, Salem, Dindugal districts and taluks of Namakkal are Tiruchengode, Kollimalai had fluoride level more than the desirable limit. The fluoride level was brought down to permissible limits with aid of the natural adsorbing agents that are physically treated. The implementation of this study makes the work more efficient and competent. There was encouraging in field water too. The promising techniques can be much beneficial by varying the initial concentration, Temperature, particle size and the PH of the aqueous solution with corresponding to the efficiency of lowering the fluoride concentration. Further, the study can be extended by working on adsorption isotherms, kinetics and equilibrium. This experimental work can even be carried out in terms of fixed bed adsorption. The depth and height of the bed can be manipulated by calculating column adsorption method. The ultimate aim of the study is to eradicate fluorosis from the endemic villages in different districts of fluoride prone area in Tamil Nadu as there is no cure for it. The only solution is the prevention of ingesting fluoride in drinking water and food.

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